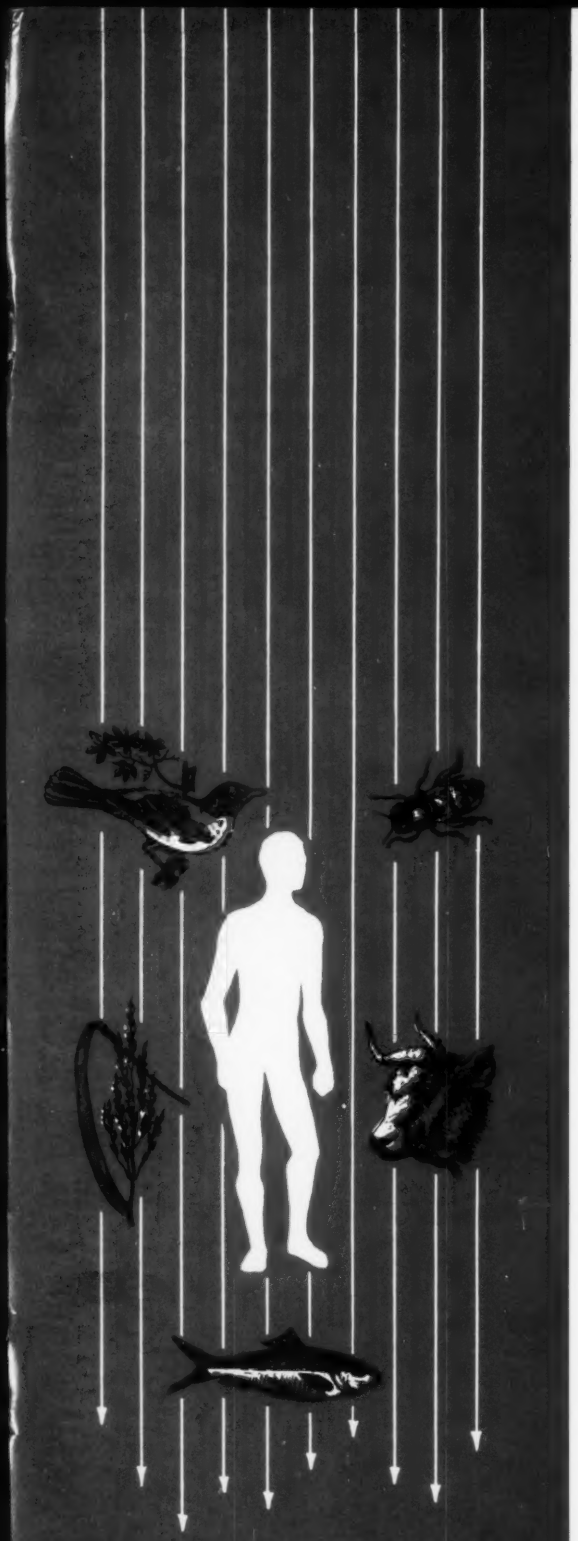

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RESIDUES IN FISH, WILDLIFE, AND ESTUARIES

Residues of Organochlorine Pesticides, Polychlorinated Biphenyls, and Mercury and Autopsy Data for Bald Eagles, 1969 and 1970 [1]

Andre A. Belisle, William L. Reichel, Louis N. Locke, Thair G. Lamont, Bernard M. Mulhern, Richard M. Prouty, Robert B. DeWolf, and Eugene Cromartie

ABSTRACT

Thirty-nine bald eagles found sick or dead in 13 States during 1969 and 1970 were analyzed for pesticide residues. Residues of DDE, dieldrin, polychlorinated biphenyls (PCB's), and mercury were detected in all bald eagle carcasses; DDD residues were detected in 38; DDT, heptachlor epoxide, and dichlorobenzophenone (DCBP) were detected less frequently. Six eagles contained possible lethal levels of dieldrin in the brain, and one contained a lethal concentration of DDE (385 ppm) in the brain together with 235 ppm of PCB's. Autopsy revealed that 18 bald eagles were illegally shot; other causes of death were impact injuries, electrocution, emaciation, and infectious diseases.

Introduction

Reichel *et al.* (*4*) presented pesticide residue data from bald eagles (*Haliaeetus leucocephalus*) collected in 1964 and 1965; a more recent report by Mulhern *et al.* (*2*) presented residue and autopsy data on bald eagles for 1966 through 1968. The purpose of this paper is to report the organochlorine pesticides, polychlorinated biphenyls (PCB's), and mercury residues and autopsy data for eagles collected in 1969 and 1970.

Sampling and Autopsy Procedures

As mentioned in previous report (*2, 4*), a systematic sampling procedure could not be used because of the relatively low populations of these birds and their protected status. Bald eagles found dead or moribund in the field are collected by Federal, State, and private cooperators, packed in dry ice, shipped air express to this laboratory, and stored intact in plastic bags at −25° C. The 13 States from which the 39 samples included in this report were collected are shown in Table 1; 28 birds were collected in 1969 and 11 in 1970. Only field specimens that were not decomposed were analyzed.

Autopsies were performed on all specimens to determine possible causes of death. Tissues for histological study were fixed in 10% formalin, embedded in paraffin, sectioned, and stained with either hematoxylin and eosin, Ziehl-Neelsen acid-fast, periodic-acid Schiff (PAS), Giesmsa, Perl's Prussian blue, or the Van Kossa stain. The entire brain was removed and placed in an acetone-rinsed glass jar, and the remaining carcass (except for skin, feet, wings, liver, and gastrointestinal tract) was

TABLE 1.—*Distribution of eagles collected, by State and year of death, 1969 and 1970*

State	Number of Eagles Collected	
	1969	1970
Florida		1
Illinois	2	1
Iowa	1	
Maine	1	
Maryland		1
Michigan	4	3
Minnesota	6	1
Missouri	5	
North Dakota	3	
Ohio	2	
South Carolina		1
Wisconsin	4	2
Virginia		1
Total	28	11

[1] From the Bureau of Sport Fisheries and Wildlife, Patuxent Wildlife Research Center, Laurel, Md. 20810.

wrapped in aluminum foil. The samples were usually processed for residue analysis within 1 week after dissection.

Analytical Procedures

The remaining carcass was ground and homogenized in a Hobart food cutter.

ORGANOCHLORINE PESTICIDES

The entire brain and a 20-g aliquot of the carcass were then extracted and cleaned up separately, using the procedure described by Mulhern *et al.* (*2*). The method consisted of Soxhlet extraction and cleanup by acetonitrile partitioning and Florisil column procedures. The eluate was concentrated and divided in half. One half was analyzed for pesticides, and the other half was set aside for PCB analysis.

The portion to be analyzed for organochlorine pesticides was streaked on a thin layer (TL) plate, and the pesticides were separated into four fractions and extracted with hot benzene (*1*). The pesticides present would be found in the following fractions: Fraction I—dieldrin, lindane, heptachlor epoxide, endrin, dichlorobenzophenone (DCBP), methoxychlor, and dicofol; Fraction II—*p,p'*- and *o,p'*-DDD; Fraction III—*p,p-'* and *o,p'*-DDT and *o,p'*-DDE; Fraction IV—*p,p'*-DDE heptachlor, aldrin, and mirex. Each fraction was analyzed by gas chromatography (GC) on a 4% SE-30/6% QF-1 column and confirmed on a 3% QF-1 or a 3% XE-60 column; instruments and operating parameters are described in Table 2.

TABLE 2.—*Gas chromatographic operating parameters using electron capture detection*

	Columns, Glass 6' × ¼" O.D.		
Instrument	Hewlett-Packard 5750	Packard	Packard
Detector	nickel 63	tritium	tritium
Liquid phase	4% SE-30/6% QF-1	3% QF-1	3% XE-60
Support	Supelcoport	Gas Chrom Q	Gas Chrom Q
Mesh size	100/120	100/120	60/80
Flow rate	argon/methane at 60 ml/min	N_2 at 40 ml/min	N_2 at 100 ml/min
Temperature	190° C	175° C	175° C

In addition to the TL zonal separations and dual-column gas chromatographic confirmation, the residues in 10% of the samples were semiquantitatively confirmed by TLC (silica gel plate—2% ethyl ether in hexane developing solvent).

Positive identification of dieldrin residues in brains of certain eagles was accomplished with the use of an LKB gas chromatograph-mass spectrometer (GC-MS) equipped with a 2% SE-30, 80/100 mesh column. Operating conditions were: flow rate—35 ml/min of helium; oven temperature—200° C; flash heater—220° C; separator—240° C; and ion source—290° C. The ionization potential was 70 ev, and accelerating voltage 3.5 kv. The sensitivity was approximately 50 ng. The mass spectra obtained from the samples were compared with spectra obtained from a dieldrin standard in respect to parent peak, Cl^{37} multiplet peaks, base peak, and fragmentation pattern.

Polychlorinated biphenyls (PCB's) with GC patterns most closely resembling Aroclor 1254® were found in all samples in Fraction IV and, in smaller amounts, in Fraction III. A previous study (*2*) showed that PCB compounds (Aroclor 1254® as reference) did not interfere in the quantitative determination of DDE unless the ratio of PCB's to DDE exceeded 2:1; samples were run on a 3% OV-17 column and quantities were measured by a disk integrator. Thus, a separate study was made to determine whether PCB compounds interfered with DDE when the DDE was quantitated by peak height using the 4% SE-30/6% QF-1 column. Standards of Aroclor 1248®, 1254®, and 1260® were each mixed with *p,p'*-DDE to obtain the follwing ratios of PCB's to DDE—1:1, 3:1, 5:1, 10:1, 15:1, and 20:1. The GC instrument parameters were the same as those used to measure DDE in the eagle samples. The results are given in Table 3.

TABLE 3.—*The interference from PCB compounds in the determination of* p,p'-*DDE*

p,p'-DDE [1]	Ratio of PCB to DDE					
	1:1	3:1	5:1	10:1	15:1	20:1
	AROCLOR 1248®					
% Recovered	100	102	100	105	105	105
% Error	—	+2	—	+5	+5	+5
	AROCLOR 1254®					
% Recovered	100	100	100	103	110	122
% Error	—	—	—	+3	+10	+22
	AROCLOR 1260®					
% Recovered	100	100	103	95	92	90
% Error	—	—	+3	−5	−8	−10

[1] Quantitation by peak height.

Using peak height, the amount of DDE recovered remained within ± 5% of true value with all three Aroclors up to a 10:1 PCB's to DDE ratio; the error increased to +10% and −8% at a 15:1 ratio of PCB's to DDE using Aroclor 1254® and 1260®, respectively.

To determine the efficiency of the analytical procedure, a pesticide recovery study was run with duck wings; 20-g aliquots of duck wing homogenates, containing trace quantities of pesticides, were spiked to contain the following: 5.0 ppm DDE, 0.2 ppm DDD, 0.1 ppm DDT, 0.2 ppm dieldrin, and 5.0 ppm Aroclor 1254®. The average recoveries from three determinations were 77% DDE, 95% DDD, 79% DDT, 85% dieldrin, and 90% PCB's.

In reporting residues in eagle samples, no corrections were made for recoveries. The lower limit of detection was arbitrarily set at 0.05 ppm; quantities <0.05 ppm were reported as trace.

PCB's

The second half of the eluate was analyzed for PCB's using the semiquantitative determination by thin layer chromatography (TLC) as described by Mulhern *et al.* (*3*) with the following modification: the sample eluate was evaporated to dryness, followed by the addition of 2 ml of oxidizing agent (1.5 g of CrO_3 in 59 ml of HOAc and 1 ml of H_2O). This agent was prepared by saturating 59 ml HOAc with a portion of the 1.5 g CrO_3; the remaining CrO_3 crystals were dissolved with 1 ml H_2O. The lower detection limit was set at 1.0 ppm; a trace value for PCB's was considered to be <1.0 ppm.

TOTAL MERCURY

Total mercury was determined by cold vapor atomic absorption. A separate 2-g portion of the homogenized carcass was digested by refluxing with sulfuric and nitric acids and then oxidized with hydrogen peroxide. Hydroxylamine hydrochloride and stannous chloride were added to the digest to reduce mercury (II) ions to mercury metal. The sample was aerated, and the mercury in the air stream passing through a gas cell was measured by atomic absorption. The lower limit of detection was approximately 0.02 ppm in a 2-g sample.

Methyl mercuric hydroxide and mercuric chloride were added to eight 2-g portions of an eagle carcass, and from 90% to 107% of the mercury was recovered.

METHYL MERCURY

Methyl mercury in ground bald eagle carcasses was determined in duplicate by the method of Jensen (Naturvardsverkets Specialanalytiska Laboratorium Lanktbrukshogskolan Uppsala, Sweden. *Unpublished method sent to Organization for Economic Cooperation and Development members, Sept. 1968*). In this procedure the lipids were extracted from a 1-g sample, wet tissue, by shaking with 30 ml methanol:ethyl ether, 3:1 mixture. After centrifuging and decanting, the remaining tissue was dried *in vacuo*, and 1 ml each of 0.5 M HBr and 0.5 M $CuBr_2$ followed by 10 ml benzene:toluene (4:1) were added. The mixture was shaken thoroughly, centrifuged, and chilled until the water layer was frozen. The organic layer was then decanted into a tube for injection into a MicroTek MT220 gas chromatograph equipped with a Ni^{63} detector on pulsed voltage operation. When chopped mallard muscle tissues spiked with 2 or 6 μg of mercury as methyl mercury hydroxide were analyzed by this procedure, 81% recoveries were obtained. Two column packings were used; one consisted of 15% Carbowax 20 M on 60/80 mesh Chromosorb G, acid washed, DCMS treated, at 185° C. Under these conditions injection of 1 ng of Hg as methyl mercury bromide produced a peak height of 10% of full-scale deflection. Some fouling of the detector was encountered with this column, and the degree of sensitivity was only fair. Thus, a second column packing, 5% Hi-Eff-10B (phenyl diethanolamine succinate) on 60/80 mesh Gas Chrom Q, was used. Operating temperatures of this column ranged from 145° to 160° C. Injection of approximately 0.4 ng Hg as methyl mercury bromide produced a peak height of 10% of full-scale deflection. This was considered to be the lower limit of reliable detection for the instrument and these operating conditions. This column packing apparently did not foul the detector, although continual clogging of the long exit tube on the detector was observed. This problem was partially alleviated by wrapping the exit tube with heating tape.

Results and Discussion

RESIDUES

The data for chlorinated pesticides, PCB's and total mercury residues found in bald eagles collected in 1969 and 1970 are summarized in Table 4. Results for eagle carcasses and brains are reported on a wet-weight basis. Medians rather than arithmetic means were used because the residue levels were asymmetrically distributed. All 39 carcass samples contained DDE, dieldrin, PCB's, and mercury; 38 contained *p,p'*-DDD.

The median value of DDE in the 1970 carcass samples was 2.6 times larger than in 1969, but both values were close to or within the median range of 4.9-16.6 ppm found for 1964-1968 (*2, 4*). For 1970, the median concentration of DDE in the brain was over 14 times greater than any median value observed since 1964; however, no trend can be established from these data because of the small number of samples collected, the wide range in DDE levels, and the absence of a systematic sampling procedure. Only two States, Minnesota and Wisconsin, were represented continuously from 1964-1970, often by one or two birds.

Significant correlations between residues of PCB's and DDE in the brain were obtained for both 1969 and 1970 samples; the correlations indicate that in the 28

samples statistically analyzed, there was an association between the levels of PCB's and DDE in the brain (Table 5). A more general inference may become possible with the analysis of future samples. The correlation between residues of PCB's and DDE in the brain could not be attributed to interference of PCB's with GC quantitation of DDE since the study of PCB interference in DDE quantitation showed only a 5% error in the amount of DDE recovered at the 10:1 level of PCB's to DDE and only one sample had a PCB's to DDE ratio greater than 10:1 (*viz* 11:1). The median value of PCB's to DDE in brains was 3.1 in 1969 and 2.2 in 1970.

The data for methyl mercury residues in 29 of the 39 carcasses are presented in Table 6; the 10 carcasses containing <1.0 ppm total mercury were not analyzed. Seven samples, which were reanalyzed in duplicate, contained less than 65% methyl mercury, presumably due to the presence of significant amounts of inorganic mercury in the soft tissues or in the bone fragments mixed with the ground carcass. The equivalent ppm mercury present as methyl mercury ranged from 0.38 to 44.56 ppm Hg. Lethal levels of methyl mercury for bald eagles have not been established.

One finding of prime interest was that pesticide poisoning possibly accounted for the death of seven bald eagles. Residues in these birds were semiquantitatively confirmed by TLC. In addition, dieldrin residues in four of these birds were analyzed and confirmed by GC-MS. Six of the eagles contained possible lethal levels of dieldrin in the brain (Table 7); two of these birds were collected in 1969 and four, in 1970. The effects of the

TABLE 4.—*Pesticide residues in bald eagles, 1969 and 1970*

[T = <0.05 ppm] [1]

Compound	Year	Residues in PPM [1]					
		Carcass			Brain		
		Median	Range	N [2]	Median	Range	N [2]
p,p'-DDE	1969	6.9	0.16-30	28	0.68	T-62	28
	1970	18	1.5-78	11	26	0.22-385	11
p,p'-DDD	1969	1.0	0.06-0.27	27	0.20	T-2.7	18
	1970	1.5	0.28-11	11	1.5	0.05-7.2	11
p,p'-DDT	1969	0.22	0.07-0.75	12	0.06	0.06	1
	1970	0.10	0.09-1.1	6	0.34	0.23-0.69	4
Dieldrin	1969	0.41	T-6.5	28	0.27	T-8.0	18
	1970	0.74	<0.10-18	11	2.0	0.23-11	10
Heptachlor epoxide	1969	0.06	T-0.35	22	0.04	0.003-0.06	6
	1970	0.10	T-0.41	9	0.36	0.06-1.0	7
Dichlorobenzophenone	1969	0.42	0.08-0.77	7	0.31	0.07-0.81	4
	1970	T	T	1	0.30	T-1.1	4
PCB Compounds	1969	10	T-150	28	2.5	T-65	28
	1970	20	4-200	11	46	T-230	11
Mercury	1969	1.5	0.52-43	28			
	1970	2.5	0.47-9.4	11			

NOTE: A total of 28 birds were collected in 1969 and 11 birds in 1970.

[1] Calculated on a wet-weight basis.

[2] Number of specimens that contained residues; the median is based on this number.

TABLE 5.—*Association between PCB and DDE residues in bald eagles, 1969 and 1970*

Year	Carcass				Brain			
	PCB/DDE		N [1]	Correlation Coefficient	PCB/DDE		N [1]	Correlation Coefficient
	Median	Range			Median	Range		
1969	1.5	0.4-10	26	0.287	3.1	0.9-11	18	[2] 0.908
1970	1.3	0.5-3.3	11	0.451	2.2	0.6-7.5	10	[3] 0.752

[1] Number of specimens that contained more than trace amounts of both PCB compounds and DDE.

[2] $P = <0.01$.

[3] $P = <0.05$.

high levels of dieldrin in the brains of the two eagles that drowned may have caused the birds to fall into the water and drown. The dieldrin levels ranged from 4.6 to 11 ppm, wet-weight basis. Experimental and field data have shown that the lowest lethal brain residue for dieldrin is about 4 or 5 ppm (*5*). The seventh eagle, an adult female from Michigan, contained 385 ppm of DDE and 6 ppm DDD in the brain, together with 235 ppm of PCB's. The level of DDE alone is well within the lethal range (*6*), but the high level of PCB's suggests that these compounds may have also contributed to death. In this study and previous studies (*2, 4*) since 1964, with the exception of one eagle of unknown sex, all bald eagles found to be possibly poisoned by dieldrin or DDT metabolites have been females. Of the 153 eagles analyzed during 1964-1970, 15 (9.8%) possibly died of dieldrin poisoning.

AUTOPSY DATA

The autopsy results for the 39 bald eagles are summarized in Table 8. Illegal shooting remained the most frequent single cause of death among the bald eagles examined in this laboratory with 46% of the birds collected during 1969-1970 having been shot.

TABLE 6.—*Mercury found in 29 bald eagle carcasses, 1969 and 1970*

Total ppm Hg [1]	Methyl Mercury Equivalent ppm Hg	% of Total Hg Found as Methyl Mercury
1.01	0.68	67.3
1.02	0.96	94.1
1.12	1.01	90.2
1.13	1.09	96.5
1.17	0.98	83.8
1.26	0.76	60.3
1.31	0.90	68.7
1.42	0.92	64.8
1.49	1.28	85.9
1.53	1.41	92.2
1.53	0.38	24.8
1.70	0.80	47.0
1.72	1.36	79.1
1.77	1.75	98.9
2.07	1.74	84.0
2.18	1.45	66.5
2.20	1.88	85.4
2.50	1.99	79.6
2.74	2.44	89.0
2.96	0.48	16.2
3.01	2.85	94.7
3.58	1.56	43.6
4.42	2.05	46.4
5.65	4.68	82.8
5.83	3.98	68.3
8.32	6.71	80.6
9.40	2.72	28.9
11.00	7.60	69.1
43.00	44.56	103.6
MEDIAN		
2.07	1.45	79.6

[1] Of the 39 samples collected, 10 contained <1.0 ppm total mercury and, thus, were not analyzed for methyl mercury.

Three eaglets were extremely emaciated; two of these were later found to have possible lethal levels of dieldrin in the brain (Table 7), and the third, a bird from Wisconsin, had multiple injuries from porcupine quills. Two quills were still embedded in the back of the eagle's oral cavity, suggesting that this eagle died from the effects of starvation and secondary bacterial infection (see below).

TABLE 7.—*Data on six suspected cases of possible dieldrin poisoning and one suspected case of DDE poisoning, 1969 and 1970*

State	Year	Age [1]	Sex	Residue in Brain (ppm)	Autopsy Findings
DIELDRIN					
Missouri	1969	Ad	F	[2] 4.6	Drowning, emaciation
Wisconsin	1969	Ad	F	[2] 6.5	Open [3]
Illinois	1970	Im	F	4.6	Open
Michigan	1970	Ad	F	[2] 4.8	Drowning
Minnesota	1970	Im	F	[2] 5.9	Emaciated; shotgun pellets around tail
Maryland	1970	Ad	F	11	Open
DDE/PCB					
Michigan	1970	Ad	F	385/235	Parasitic enteritis

[1] Ad = adult, Im = immature.

[2] Dieldrin analyzed and confirmed by gas chromatography-mass spectrometry.

[3] Open = no diagnosis could be made on the basis of autopsy findings.

TABLE 8.—*Probable causes of bald eagle mortality, 1969 and 1970*

Cause of Death	Number of Eagles
Shot	18
Dieldrin [1]	6
DDE [1]	1
Impact	4
Electrocution	2
Emaciation	1
Nephrosis	1
Streptococcal infection	1
Avian cholera	1
Trapping injuries	1
Open	3
Total	39

[1] See Table 7 for details.

One eagle from Michigan had an old trapping injury which had become secondarily infected and resulted in a fibrinous pericarditis. A pure culture of a gram-positive rod, subsequently identified as a *Lactobacillus*, was isolated from the pericardium. This isolate of *Lactobacillus* was noninfectious to laboratory mice and domestic pigeons and probably should be regarded as a postmortem contaminant.

Although *Pseudomonas aeruginosa* was isolated from the kidney and from an old infected bullet leg wound of an eagle from Wisconsin, the cause of death was a more recent gunshot wound which produced massive hemorrhage into the pericardial sac.

Pasteurella multocida, the causative organism of avian chlorea, was isolated from three eagles. The first isolation was from an adult male from Ohio and apparently represents a frank case of avian chlorea. The second isolation of *P. multocida* was from the heart of the emaciated eagle which had been injured by porcupine quills; the third isolation was from the liver of an eagle shot in Florida.

Several parasitic conditions were observed in these 39 eagles. Schizonts of *Leucocytozoon* were found in the hearts of an eagle from Maryland and one from Illinois; both eagles had possible lethal levels of dieldrin in the brain (Table 7). Another eagle from Michigan, subsequently found to have high levels of DDE and PCB's in the brain, had a severe enteritis caused by a large number of as yet unidentified flukes; although the enteritis was severe, it was not regarded as the cause of the eagle's death.

Conclusion

Since the bald eagle is located at the top of food chains and is the final recipient of environmental pollutants, the presence of PCB's, DDE, dieldrin, and mercury in all 39 samples from 13 States, demonstrates continued widespread environmental contamination by these compounds.

Acknowledgment

Appreciation is extended to J. O. Knisley and P. Jones who assisted in the autopsies and dissections of eagles, to L. T. Young who prepared sections for histological examination, and to the cooperators from many States and organizations who submitted eagles for analysis. Special acknowledgment is due to Dr. R. Shillinger and his staff (Animal Health Laboratories, Maryland Department of Agriculture, College Park, Md.) for the identification of the bacterial isolates and to the National Animal Disease Laboratory, USDA, Ames, Iowa, for identification of the *Lactobacillus* isolate.

See Appendix for chemical names of compounds discussed in this paper.

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DDT, DDE, and Polychlorinated Biphenyls in Biota From the Gulf of Mexico and Caribbean Sea—1971

C. S. Giam[1], A. R. Hanks[2], R. L. Richardson[1], W. M. Sackett[3], and M. K. Wong[4]

ABSTRACT

Residue levels of DDT, DDE, and PCB's were determined in various species of fish, shrimp, crabs, and other biota from the Gulf of Mexico and Carribean Sea. Samples were collected from the Gulf during two Gulf-wide cruises in May and October 1971 and from part of the Carribean Sea during the October cruise. DDT, DDE, and PCB's were found widely distributed in all biota; however, samples from coastal areas generally had higher levels than samples from the open waters.

Introduction

The occurrence of DDT and its metabolites and polychlorinated biphenyls (PCB's) in fish and wildlife is of current interest; however, data concerning the levels of these compounds in organisms from the open ocean are scarce (*1-5*).

During 1971 and 1972 about 50 scientists are participating in an intensive study program sponsored by the International Decade for Ocean Exploration (National Science Foundation) to identify problems related to oceanic environmental quality. This paper reports some of the results of this study, i.e., the concentrations of DDT, DDE, and PCB's in fish and other marine organisms collected in the Gulf of Mexico and the Caribbean Sea in May and October 1971. These "baseline concentrations" should be useful as comparison data to investigators working in estuaries around the Gulf of Mexico. Fig. 1 shows the locations where samples were collected.

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The Gulf of Mexico receives runoff from approximately two-thirds of the United States and one-half of Mexico. This large amount of runoff with its high load of pollutants is swept generally westward and trapped in the western Gulf where waters remain possibly as long as 100 years. The primary flushing mechanism is exchange in the eastern Gulf with the loop current which passes quickly through the Yucatan Strait and out through the Straits of Florida. Because of the characteristics of this unique system, a buildup in concentration of man-made toxic materials is possible in the western Gulf, and baseline concentrations reported here together with future analyses should provide an early indication of this.

Sampling and Analytical Procedures

Samples were collected by the scientific party aboard cruises 71-A-5 (in May 1971) and 71-A-12 (in October 1971) of the R/V Alaminos, the oceanography vessel of Texas A&M University. Most samples were obtained using nets, but a few tuna and shark were caught by hook and line. Smaller samples were transferred immediately to glass mason jars with caps lined with aluminum foil and frozen until analysis. The jars and foil had been pre-washed with absolute ethanol which was free of any chlorinated hydrocarbons. Appropriate organs and muscle samples were taken from the larger fish, placed in mason jars, and frozen until analysis.

Small fish, shrimp, crabs, and other crustaceans were analyzed whole as composites of two or more fish or six crustaceans. Generally 50- to 100-g subsamples were taken from each composite for analysis. Subsamples of muscle tissue and organs analyzed generally weighed 50- to 100-g. The extraction and cleanup procedure used was that described in the "Pesticide Analytical Manual"

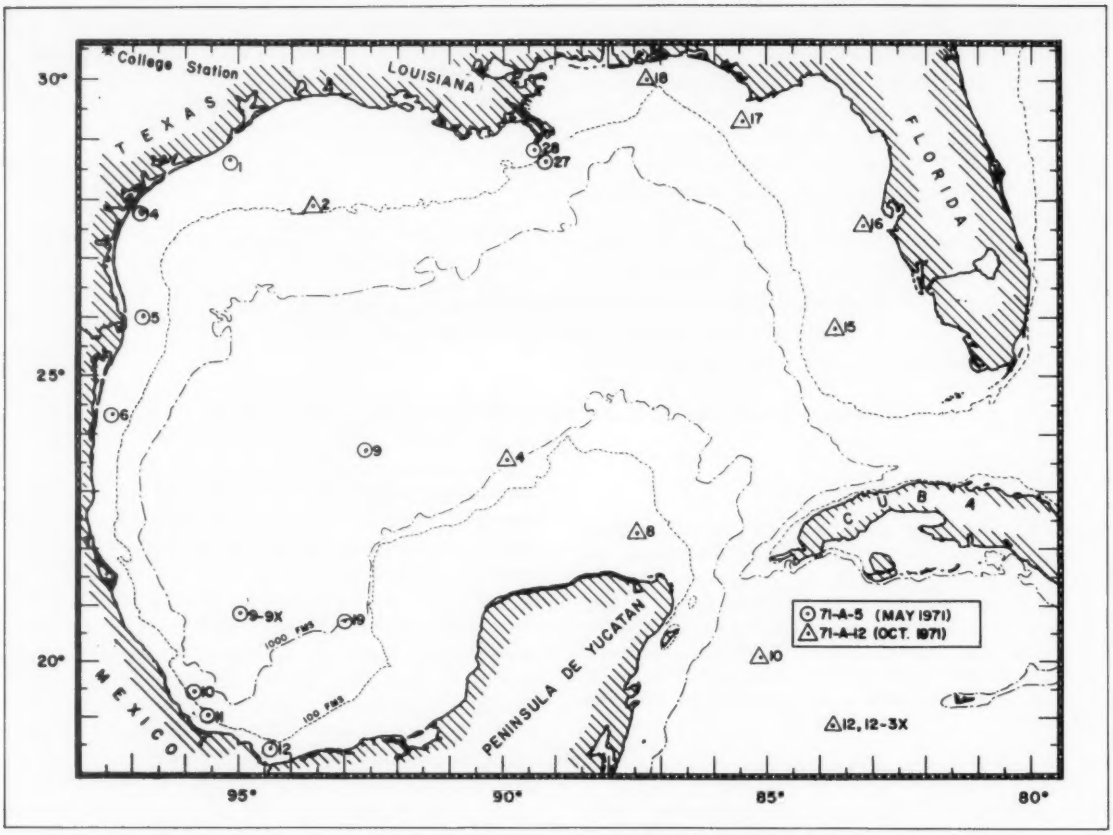

FIGURE 1.—*Sampling stations in the Gulf of Mexico and Caribbean Sea—May and October 1971*

of the U. S. Food and Drug Administration (*6*). The final residue extracts were adjusted to a suitable volume (between 2 and 10 ml) for gas chromatographic analysis. No attempt was made to concentrate the eluate to less than 2 ml, and not more than 10 μl of extract was injected into the chromatographic column.

A Tracor gas chromatograph (Model MT 220) equipped with a ^{63}Ni electron capture detector and U-shaped glass columns, 6 ft x ¼ inch, o.d., and packed with 5% DC-200 on 80/100 mesh HP Chromosorb W was used. Columns packed with 5% OV-1 on 80/100 mesh HP Chromosorb W and a 6% mixture of OV-17 and QF-1 (in the ratio of 7:9) on 80/100 mesh HP Chromosorb W were also employed for further characterization. Nitrogen was used as the carrier gas at a flow rate of 60 cc/min. The injector, oven, and detector temperatures were 225° C, 200° C, and 275° C, respectively.

Identification of PCB's as commercial Aroclor® formulations was based on good matching of the sample peaks with those of standard Aroclor® mixtures. Quantification was carried out when at least 50% of the peaks from a sample chromatogram matched peaks of the Arcoclor® formulation; three different columns were used for matching the arrays of Aroclor®. In instances where the concentrations of PCB's were so high that they interfered with the quantification of DDT and DDE, separations of the PCB's from DDT and its metabolites were performed using a silica gel column (*7*). The presence of DDT and PCB's was further confirmed by the disappearance of the *p,p'*-DDT peak (and a corresponding increase in the *p,p'*-DDE peak) in the chromatograms of sample extract after alkaline alcoholysis treatment (*8*). The PCB peaks were not affected.

Percent recovery studies were performed using spiked liver and muscle samples. Recovery was 85% or better for all compounds identified in this study. (The group performing these analyses took part in a national and international cooperative analysis of chlorinated hydrocarbon insecticides and PCB's in marine samples, and their results were in excellent agreement with results from other laboratories.) The sensitivity of detection,

depending on the weight of the sample, ranged from 0.1 to 0.3 μg/kg wet weight for DDT and DDE and 1 to 3 μg/kg for PCB's. Results were not corrected for percent recovery.

Results and Discussion

Results of analysis of samples from the Gulf of Mexico and the Caribbean Sea are given in Tables 1 and 2. DDT, DDE, and the PCB's were detected in nearly all the samples analyzed, indicating that these compounds were widely distributed in the Gulf and Caribbean Sea; however, the levels were generally low, as may be expected in open ocean biota (*5, 9*). *P, p'*-DDD was not detected, and the analytical procedures excluded other organochlorine insecticides.

The levels of residues in marine organisms varied appreciably, but because the number of samples analyzed from each location was small, no firm conclusions can be made at this time. Certain general trends were evident, however, from the results of these analyses: (1) The samples from coastal areas, regardless of species, generally had higher levels of DDT, DDE and PCB's than samples from open waters. For example, samples obtained in May 1971 from Stations 27 and 28—two sites near the Mississippi Delta, a highly polluted area—showed relatively higher concentrations. (2) The ratios of DDE to DDT varied widely between samples. A few samples from the coastal areas, i.e., shrimp collected at Stations 4, 6, 10, and 28 and other crustaceans from Station 27, showed rather high levels of DDT but relatively low levels of DDE (Table 1). Residue data from these specific samples may be fortuitous or may indicate that most of the DDT had not yet been metabolized. (3) In the livers obtained from larger fish (Table 2), concentrations of DDE were generally higher than DDT, possibly indicating the capability of this organ to metabolize and degrade DDT. (4) In individual fish (Table 2), the liver generally had the highest levels of DDT, DDE, and PCB's and muscle tissue the lowest; appreciable concentrations were also detected in the gonads and the digestive tract.

Acknowledgments

We gratefully acknowledge the National Science Foundation (International Decade of Ocean Exploration program) for financial support of this work, Mr. W. Dill for identification of organisms analyzed in this study, and Mr. Jerry Burke of the Food and Drug Administration and Dr. George Harvey of the Woods Hole Oceanographic Institute for intercalibration samples.

[1] See Appendix for chemical names of compounds discussed in this paper.

TABLE 1.—*DDT, DDE, and PCB residues in biota from the Gulf of Mexico and Caribbean Sea—May and October 1971*

Month of Collection (1971)	Sampling Station (See Fig. 1)	Location	Sample [1]	Residues in μg/kg, wet/weight basis			
				p,p'-DDT	*p,p'*-DDE	Total DDT	PCB's [2]
May	1	Lat. 28°43.1′ Long. 95°08.4′	Flounder (*Syacium ounteri*)	84	10	94	32
			Squid	65	4.6	70	[3]
	4	Lat. 27°47.1′ Long. 96°49.6′	Crabs (*Callinectes sp.*)	1.8	7.4	9	[4] 17
			Fish (Unidentified)	141	18	159	[4] 53
			Flounder (*Syacium papillosum*)	21	14	35	36
			Sea pansy (Order Pennatulacea)	128	161	289	[4] 850
			Shrimp (Family Penaeidae)	26	7.0	33	[3]
	5	Lat. 26°02.8′ Long. 96°48.5′	Fish (*Paraques acuminatus*)	20	6.6	27	27
			Flounder (*Syacium papillosum*)	[5]	[5]	[5]	59
	6	Lat. 24°23.4′ Long. 97°23.9′	Flounder (*Syacium ounteri*)	3.6	3.3	7	34
			Shrimp (Family Penaeidae)	134	18	152	[3]

TABLE 1. —*DDT, DDE, and PCB residues in biota from the Gulf of Mexico and Caribbean Sea— May and October 1971*—Continued

Month of Collection (1971)	Sampling Station (See Fig. 1)	Location	Sample [1]	Residues in µg/kg, wet/weight basis			
				p,p'-DDT	*p,p'*-DDE	Total DDT	PCB's [2]
May	9-9X	Lat. 20°53.5′ Long. 94°59.0′	Flying fish (Family Exocoetidae)	7.2	4.5	12	20
	10	Lat. 19°28.0′ Long. 95°51.0′	Colonial tunicate	188	8.7	197	[4] 139
			Shrimp (Family Penaeidae)	154	11	165	[3]
	11	Lat. 19°02.0′ Long. 95°35.9′	Fish *(Peristedion oracile)*	16	16	32	[4] 54
			Fish *(Serranus atrobranchus)*	33	8	41	[3]
			Fish *(Saurida brasiliensus)*	111	7	118	68
			Fish *(Halicutichthys aculeatus)*	78	4.8	83	[4] 64
			Fish *(Trichoosetta ventralis)*	8.5	5.2	14	[3]
	19	Lat. 20°44.0′ Long. 92°50.0′	Squirrel fish *(Holocentrus sp.)*	86	5.6	92	[4] 150
	27	Lat. 28°40.9′ Long. 89°10.0′	Crustacean *(Aristacus antillensia)*	86	16	102	151
			Crustacean *(Nephropsis aculeate)*	151	6.0	157	22
			Fish *(Benthodesmus atlanticus)*	91	4.3	95	36
			Fish (Unidentified)	33	7.3	40	56
			Holothuroids	[5]	[5]	[5]	[4] 8
	28	Lat. 28°21.7′ Long. 90°14.0′	Bat fish	12	65	77	527
			Croakers *(Micropogon undulatus)*	[5]	11	11	50
			Shrimp *(Parapenaeus longirostris)*	304	34	338	167
October	10	Lat. 20°05.4′ Long. 85°07.7′	Flying fish (Family Exocoetidae)	5.1	4.6	10	26
	12-3X	Lat. 18°54.1′ Long. 83°44.4′	Flying fish (Family Exocoetidae)	46	19	65	[3]
	15	Lat. 25°49′ Long. 83°43′	Fish *(Synodus intermedius)*	4.5	10	15	14
	16	Lat. 27°34′ Long. 83°10′	Flying fish (Family Exocoetidae)	18	12	30	65
	17	Lat. 29°19.5′ Long. 85°28.0′	Rock shrimp	1.0	2.1	3	[3]
	18	Lat. 30°00.5′ Long. 87°17.5′	Rock shrimp	5.2	2.7	8	6
			Squid	4.6	4.3	9	40

[1] Represents 50- to 100-g subsamples from a composite of two or more whole fish or six whole crustaceans/other invertebrates.
[2] Calculated as Aroclor 1260® unless otherwise indicated.
[3] Not estimated because of insufficient number of peaks on the chromatogram to characterize PCB formulations.
[4] Calculated as Aroclor 1254®.
[5] Not analyzed due to negligible concentrations of DDT and interference by PCB peaks.

TABLE 2.—*DDT, DDE, and PCB residues in organs and muscle tissue of fish from the Gulf of Mexico and Caribbean Sea—May and October 1971*

Month of Collection (1971)	Sampling Station (See Fig. 1)	Location	Fish Collected	Tissue or Organ Sampled [1]	Residues in μg/kg, wet-weight basis			
					p,p'-DDT	*p,p'*-DDE	Total DDT	PCB's [2]
May	5	Lat. 26°02.8′ Long. 96°48.5′	Shark *(Carcharinus falciformis)*	liver	200	499	699	1300
	9	Lat. 23°45.8′ Long. 92°37.4′	White tip shark *(Pterolamniops longimanus)*	gut	53	62	115	58
				gonads	188	60	248	74
				muscle	15	15	30	32
				liver	406	1100	1506	536
	12	Lat. 18°27.5′ Long 94°24.0′	King mackerel *(Scomberomous cavalla)*	gut	15	14	29	90
				gonads	3.0	15	18	56
				muscle	17	7.4	24	34
				liver	72	51	123	83
			Tuna *(Euthynnus alleteratus)*	gut	14	13	27	76
				muscle	44	31	75	58
				liver	45	39	84	59
	19	Lat. 20°44.0′ Long. 92°50.0′	Parrot fish *(Holichoeres radiatus)*	liver	36	[3]	36	[4] 284
			Red snapper *(Lutjanus aya)*	liver	3.9	9.5	13	18
				gonads	34	6.1	40	16
			Trigger fish *(Canthidermis sufflamen)*	liver	83	52	135	[5]
			Yellow tailed snapper *(Ocyurus chrysurus)*	liver	10	19	29	43
				gonads	37	7.4	44	22
				gut	56	3.7	60	9
October	2	Lat. 27°54.5′ Long. 93°36.0′	Jack *(Thunnus atlanticus)*	muscle	36	13	49	43
	4	Lat. 23°33.4′ Long. 89°54.5′	Trigger fish *(Canthidermis sufflamen)*	muscle	2.7	1.9	5	<1
	8	Lat. 22°16.4′ Long. 87°27.3′	Tuna *(Euthynnus alleteratus)*	muscle	8.2	36	44	36
				gonads	4.4	8.4	13	35
				liver	40	111	151	153
	12	Lat. 18°54.1′ Long. 83°44.4′	Barracuda *(Sohyraana barracuda)*	muscle	4.0	4.2	8	9
				liver	16	26	42	57
			Fish *(Haemulon plumieri)*	muscle	2.5	1.2	4	<1
			Shark *(Carcharhinus springeri)*	muscle	[3]	1.2	1	8
				liver	44	116	160	310
			Trigger fish *(Balistes vetula)*	muscle	1.4	0.6	2	<1
			Trigger fish *(Canthidermis sufflamen)*	muscle	1.7	0.9	3	<1

[1] Represents 50 to 100 g of organ or muscle samples.
[2] Calculated as Aroclor 1260® unless otherwise indicated.
[3] Not analyzed due to negligible concentrations and interference by PCB peaks.
[4] Calculated as Aroclor 1254®.
[5] Not estimated because of insufficient number of peaks on the chromatogram to characterize PCB formulations.

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Mercury Residues in Fish, 1969-1970—National Pesticide Monitoring Program [1]

Croswell Henderson, Anthony Inglis[2], and Wendell L. Johnson

ABSTRACT

As part of the fish monitoring program conducted by the Bureau of Sport Fisheries and Wildlife since 1967, composite fish samples collected during the fall of 1969 and 1970 were analyzed for mercury. Three composite samples, each of a different species and consisting of 3-5 adult fish, were collected at each of 50 monitoring stations in 1969; similarly, three composite samples and in most cases a replicate sample of one of the species were collected at each of 100 stations in 1970. Stations were located on major rivers and lakes throughout the United States. Total mercury residues equal to or exceeding the sensitivity level of 0.05 ppm were found in 129 of the 145 samples in 1969 and 373 of the 393 samples in 1970. Values ranged from <0.05 to 1.25 ppm in 1969 samples and from <0.05 to 1.80 ppm in 1970 samples. Analyses by two different laboratories of 40 selected samples from the 1970 collection gave comparable results. Analyses of 24 selected 1970 samples indicated that 90% or more of the mercury in fish was in the form of methyl mercury.

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Introduction

A nationwide monitoring program to determine pesticide residue levels in fish has been conducted by the Bureau of Sport Fisheries and Wildlife each year since 1967. Composite fish samples collected from 50 stations during the first 2 years of the program (1967, 1968) were analyzed for whole body residues of 11 organochlorine insecticides; these results were reported in a previous issue of this Journal (*3*). In 1969, fish were collected from the same 50 stations, and analyses were expanded to also include lipids, polychlorinated biphenyls (PCB's), and mercury; results for lipids, organochlorine insecticides and PCB's were reported by Henderson, Inglis, and Johnson (*4*). In 1970, the number of sampling stations was increased to 100 and analyses again included organochlorine insecticides, PCB's, lipids, and mercury.

This report presents the data on mercury residues in fish collected from 50 stations during 1969 and from 100 stations during 1970. Common names of fishes as designated by the American Fisheries Society (*1*) are used throughout this report.

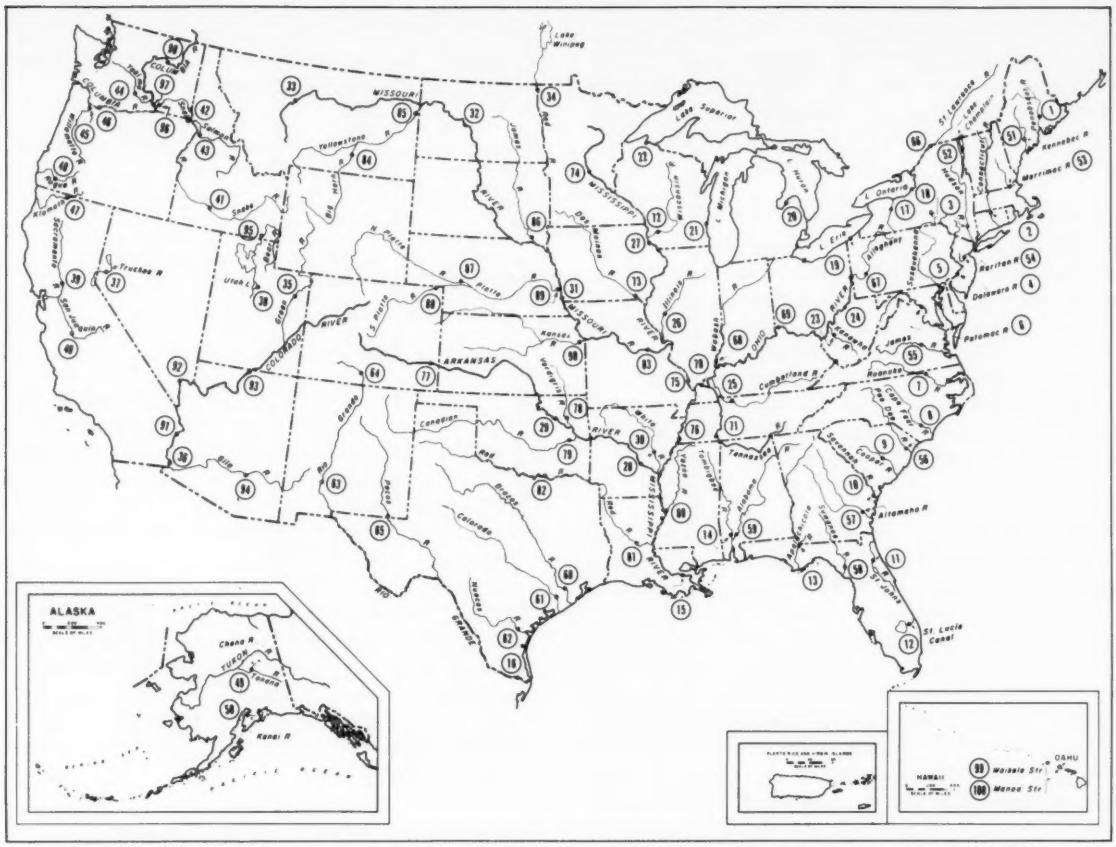

FIGURE 1.—*Locations of fish sampling stations, National Pesticide Monitoring Program—1969 and 1970*

Methods

FISH COLLECTIONS

The locations of sampling stations are shown in Fig. 1 and listed in Table 1. Fish were collected at Stations 1-50 in both 1969 and 1970, but at Stations 51-100 in 1970 only.

As in previous collections, three composite samples, each of a different species and consisting of 3-5 adult fish of uniform size for each species, were collected at each station. In 1970, a replicate composite sample of one of the three species was also collected in order to determine possible variation in residue levels in similar samples from the same station. A special effort has been made to collect the same species each year.

Fish collections were made by biologists from Fishery Services and other Divisions of the Bureau of Sport Fisheries and Wildlife with considerable assistance from State conservation agencies. Fish were collected by various means including seines, gill nets, traps, hook and line, electrofishing, etc. The use of fish toxicants was not permitted. Collections were made once each year, usually in September, October, or November.

Each composite sample was wrapped in aluminum foil, frozen, packed in dry ice, and shipped to a laboratory for whole body residue analyses. Accompanying the shipment was a legend showing location, date collected, name of collector, collection method, species of fish, and the length, weight, and estimated age of each fish in each composite sample.

LABORATORY ANALYSES

A commercial laboratory (designated Laboratory C) conducted total mercury analyses on all of the 1969 and 1970 samples. The same laboratory conducted methyl mercury analyses on 24 selected samples from the 1970

fish collection. Subsamples of 40 selected homogenates from the 1970 collection were sent to another laboratory (designated Laboratory H) for total mercury analyses in order to cross-check or further confirm the results from Laboratory C. Analytical methods as furnished by participating laboratories are as follows:

Laboratory C—Analyses for Total Mercury

Each composite sample was thawed, cut into small pieces, and ground in a Hobart food chopper until homogenized. An aliquot sample was removed for mercury analysis. The method used is described in a report by the Joint Mercury Residues Panel (*7*), modified by atomic absorption spectrometry with the boat technique for 1969 samples and the cold vapor technique for 1970 samples. The procedures for digestion and analysis by the cold vapor technique were as follows:

A 10-g portion of the fish homogenate was transferred to a 1-liter round-bottom flask using little or no water; several glass beads were added. The flask was placed in a mantle and condensers and tap funnel inserted; 25 ml of a sulfuric-nitric acid mixture (4:1) was carefully introduced through the tap funnel (over approximately 10 minutes). The sample was heated slowly, so that the reaction would not become violent, for approximately ½ to ¾ of an hour increasing the heat until full heat was reached. When necessary, small amounts of nitric acid were added to prevent carbonization. After refluxing for one hour, the sample was cooled to room temperature. When the digest was cool, the condenser and tap funnel were disconnected and the digest was transfrered quantitatively with ice water to a 100-ml volumetric flask. The flask was stoppered and allowed to come to room temperature. The digest was made to volume, mixed, and analyzed on an atomic absorption spectrophotometer.

For atomic absorption analysis, 50 ml of a reducing solution was transferred into a 300-ml Erlenmeyer flask. The reducing solution was made up with 5.0 g of NaCl, 10 g of hydroxylamine hydrochloride, 20 g of stannous chloride in 20% H_2SO_4, and diluted to 1 liter with 20% H_2SO_4. Additional 20% sulfuric acid was added to make a total volume of 75 ml with the amount of sample to be added. The sample was added with a pipet by draining down the side of the flask, the flask stoppered rapidly, and then stirred vigorously with a magnetic stirrer (teflon bar) for exactly 30 seconds. The stirrer was shut off and the teflon bar allowed to stop; then the air pump was turned on to force mercury through the cell. To clean out the cell, the air flow was reversed between each sample analysis.

To establish a standard curve, an approximately 20-fold range of standards was used starting with 0.010 μg of mercury. The standard curve was plotted using peak height versus micrograms of mercury.

Analyses were performed on a Perkin-Elmer atomic absorption spectrometer, model 303, and a Perkin-Elmer recorder, model 304, with the following conditions:

Wavelength—2537 Angstroms (254 Setting—303).
Slit—3 mm, 20 Angstroms (5 Setting—303)
Range—UV
Source—Mercury hollow cathode lamp
Air—3 liters per minute
Recorder noise suppression—1, expansion—3×

The same digestion procedure as above was used for the boat method. Mercury was extracted from the acid solution into a dithizone-chloroform solution with two 5-ml extractions. Extractions were placed into a small tantalum boat and chloroform vaporized off. The boat was then placed directly into the flame, and the mercury vaporized.

Fourteen samples analyzed by both the boat and cold vapor techniques gave comparable results. However, the cold vapor technique was found to be more rapid and to have greater sensitivity with less interference. Recovery experiments showed an average recovery rate of 97%. The results were not corrected for recovery; sensitivity was reported as 0.05 ppm.

Laboratory C—Analyses for Methyl Mercury

Methyl mercury was determined by the procedure developed by Kamps and McMahon (*8*).

Laboratory H—Analyses for Total Mercury

The digestion method used was patterned after that reported by the Analytical Methods Committee (*2*); the flameless atomic absorption method was quite similar to that reported by others (*6*). A description of the methods follows:

For digestion, up to 10 g (wet weight) of sample was placed in a 250-ml flat-bottom flask with 3 boiling beads. This was placed under a condenser, and 10 ml of HNO_3 added through the condenser. When necessary, the sample was warmed slightly to start the reaction, but heating was discontinued when foaming started. When the initial reaction was finished, the sample had been dissolved. The flasks were allowed to cool slightly, and then 10 ml of 1:1 H_2SO_4:HNO_3 was added. The sample was heated slowly, without boiling, until refluxed, then refluxed for 3 hours. The flask was cooled until just warm to touch, and 10 ml of 30% H_2O_2 added in 1-ml increments, waiting between additions for foaming

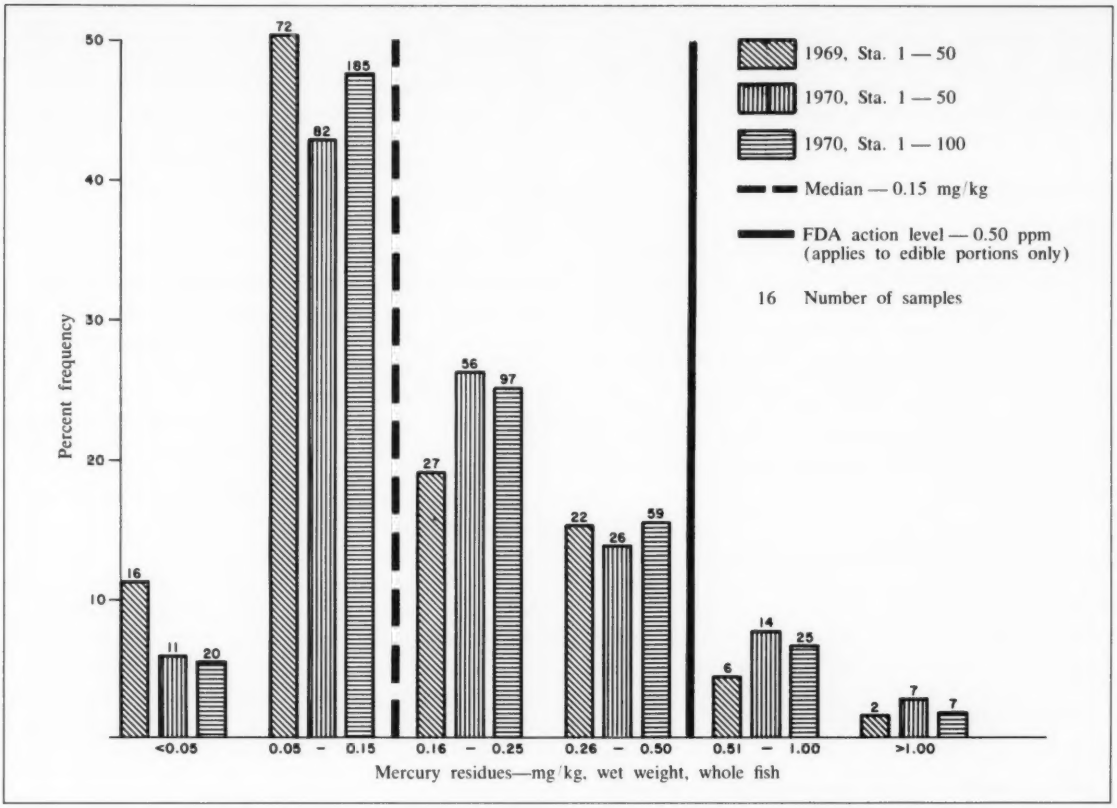

FIGURE 2.—*Mercury residues in fish by frequency of occurrence—1969 and 1970*

to stop. If no foaming occurred on the first addition, the sample was heated slightly until foaming began. After all 10 ml had been added, the sample was brought to a boil, and boiled for 1 hour (by then most of the brown NO_2 fumes had gone). The sample was chilled in ice water, and the condensers washed down with 50 ml of redistilled water; the sample was removed from the condensers after the fat solidified. At this point, the sample was analyzed or covered and left in the refrigerator overnight.

For atomic absorption analysis, the cold digestion sample was filtered through glass wool into a 100-ml graduated cylinder, and the flask and filter were washed with enough redistilled water to make the volume up to 100 ml. A 1-ml or some other suitable aliquot was removed and added to a gas bubbling flask containing sufficient 1N H_2SO_4 to make a total volume of 20 ml. Two milliliters of the reductant solution (100 ml of H_2SO_4, 5 g of NaCl, 5.3 g of NH_2OH:HCl, and 20 g of $SnCl_2$:$2H_2O$, made to 1 liter) was added, the flask closed, the chart recorder started, the gas flow turned on, and the maximum pen deflection on chart noted. Then, the gas flow was turned off and a suitable quantity of standard (from 0.1 ppm Hg in 1N H_2SO_4) added. The gas was turned on and measured as before, and the nanograms of Hg were plotted against the peak area on linear graph paper.

Analyses were performed on a Jarrell-Ash Model 82 atomic absorption spectrometer equipped with a 25-cm quartz cell.

Results and Discussion

FISH COLLECTIONS

A total of 145 composite samples were collected from the 50 sampling stations in 1969 and 393 samples from the 100 stations in 1970. Fifty-five species of fish were represented in the collections. Some species such as carp, channel catfish, and largemouth bass were collected at many different stations. For example, samples of carp were obtained from 56, channel catfish from

34, and largemouth bass from 33 of the 100 stations in 1970. Many of the species, however, were collected at only 1 or 2 of the 100 stations. Generally, collectors were successful in obtaining the same species at a station in both 1969 and 1970 collections.

RESIDUE LEVELS IN FISH

Total mercury residue levels for each composite sample and average values at each station for both 1969 and 1970 collections are shown in Table 1. All values are reported as ppm (mg/kg), wet-weight, whole fish. Also shown in Table 1 are station locations and species of fish; the number and the average length and weight of fish in the composite samples collected in both years are included. The analyses for mercury in 1969 samples were conducted on subsamples of fish homogenates that had been prepared primarily for organochlorine insecticide analyses.

In order to make the 1969 and 1970 data more comparable, the 1970 average residue values were computed as follows: the replicate samples of the same species were averaged and this average used with the values for the other species to compute the final average. Values less than the sensitivity level of 0.05 ppm were considered to be zero in computing averages.

Mercury residues equal to or exceeding the sensitivity limit were found in 129 of the 145 samples in 1969 and 373 of the 393 samples in 1970. Values ranged from <0.05 to 1.25 ppm mercury in 1969 samples and from <0.05 to 1.80 ppm Hg in 1970 samples.

While high residue levels were found in fish from some waters in most drainage basins, these high levels appeared to occur more frequently in samples from Atlantic Coastal streams and the Columbia River system (Fig. 1). Lowest levels were found in fish from Alaskan streams, the Colorado River system, and Mississippi River tributaries in the Great Plains region.

Total mercury residues exceeding the Food and Drug Administration action level of 0.5 ppm, set for edible portions of the fish only, were found in 8 samples from 8 of the 50 stations in 1969 and in 32 samples from 21 of 100 stations in 1970. Average values exceeded 0.5 ppm at 3 stations in 1969 and 6 stations in 1970.

The frequency of occurrence of various residue levels is shown in Fig. 2. The range of values was comparable for 1969 and 1970 samples with the median residue level the same (0.15 ppm) for both sampling periods.

High mercury levels occurred much more often in some species of fish than in others. With few exceptions, the highest levels were found in predatory species near the top of the food chain such as bass, perch, and squawfish. Generally, the residue results for the two composite samples of the same species collected at each station in 1970 were in close agreement indicating no great variation in similar samples.

CONFIRMATORY ANALYSIS AND METHYL MERCURY

The results of the initial analyses by Laboratory C of 40 selected samples from the 1970 collections and the analyses of subsamples of the same homogenates by Laboratory H for total mercury are shown in Table 2. Also shown are results of analyses for methyl mercury reported as mg/kg of Hg on 24 selected samples by Laboratory C. The samples selected for methyl mercury analyses were among those having the highest total mercury residues.

The total mercury residue results for both laboratories are in very close agreement, although slightly different methods were used.

From a comparison of the total mercury and methyl mercury results, it appears that at least 90% and possibly more of the mercury in fish is methyl mercury.

The significance of the mercury residue results with respect to possible adverse effects on fish and wildlife is unknown at the present time. Research is underway to determine possible correlation of residue levels in tissues with possible effects such as mortality, reproduction, etc. From the standpoint of the use of fish for human food, it may be significant that residue levels in some samples exceeded the FDA action level of 0.5 ppm. It is also possible that such levels may have an adverse effect on fish-eating birds or other animals.

Monitoring for mercury and other residues in fish is a continuing program (5). Fish samples were collected at the same 100 sampling stations again in the fall of 1971 and are presently being analyzed for residues of mercury, other metals, organochlorine insecticides, and PCB's.

Acknowledgment

We greatly appreciate the help of the Bureau of Sport Fisheries and Wildlife biologists and fishery personnel in many of the States for collecting samples for the monitoring program. Without their assistance, such a program would not have been possible.

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TABLE 1.—*Mercury residues in fish, 1969 and 1970*

Station Number and Location		Species	1970				1969			
			No. of Fish	Average Size		Total Mercury (PPM)[1]	No. of Fish	Average Size		Total Mercury (PPM)[1]
				Length (Inches)	Weight (Lb)			Length (Inches)	Weight (Lb)	
ATLANTIC COASTAL STREAMS										
#1	Stillwater River Old Town, Maine	White sucker	5	15.1	1.3	.43	5	15.0	1.5	.23
		White sucker (R)	5	15.3	1.3	.60				
		Yellow perch	5	7.9	0.2	.29	5	7.7	0.2	.48
		Chain pickerel	4	16.4	1.0	.64	5	13.7	0.5	.42
						Avg. .48				Avg. .38
#51	Kennebec River Hinckley, Maine	Yellow perch	5	9.3	0.4	1.20				
		Yellow perch (R)	5	9.0	0.4	.71				
		White perch	2	8.9	0.5	.38				
		Smallmouth bass	2	11.3	0.9	.64				
						Avg. .66				
#52	Lake Champlain Burlington, Vt.	Pumpkinseed	5	6.5	0.3	.38				
		Yellow perch	5	8.6	0.3	.27				
		Yellow perch (R)	5	9.4	0.5	.49				
		Chain pickerel	5	16.2	1.1	.47				
						Avg. .41				
#53	Merrimac River Lowell, Mass.	White sucker	5	12.0	0.6	.27				
		White sucker (R)	5	10.4	0.5	.28				
		Pumpkinseed	5	5.6	0.1	.15				
		Yellow perch	3	7.9	0.2	.33				
						Avg. .25				
#2	Connecticut River Windsor Locks, Conn.	White catfish	5	12.6	0.9	.22	5	12.0	0.9	.14
		White catfish (R)	5	12.9	1.0	.18				
		Yellow perch	2	10.5	0.6	.30	3	8.8	0.4	.13
		White perch	5	9.9	0.6	.51	5	10.0	0.6	.80
						Avg. .34				Avg. 36
#3	Hudson River Poughkeepsie, N. Y.	Goldfish	5	8.4	0.5	.07	2	11.7	1.5	.16
		Goldfish (R)	5	10.5	1.0	.11				
		Largemouth bass	4	12.8	1.3	.19	5	9.2	0.5	.25
		Pumpkinseed	5	6.2	0.2	.10	5	6.2	0.2	.19
						Avg. .13				Avg. .20

TABLE 1.—*Mercury residues in fish, 1969 and 1970*—Continued

Station Number and Location		Species	1970				1969			
			No. of Fish	Average Size		Total Mercury (PPM)[1]	No. of Fish	Average Size		Total Mercury (PPM)[1]
				Length (Inches)	Weight (Lb)			Length (Inches)	Weight (Lb)	
ATLANTIC COASTAL STREAMS—Continued										
#54	Raritan River Highland Park, N. J.	Golden shiner	5	5.9	0.1	.08				
		White perch	5	9.1	0.5	.28				
		White perch (R)	5	8.3	0.3	.29				
		Rock bass	3	6.9	0.2	.25				
						Avg. .21				
#4	Delaware River Camden, N. J.	White sucker	5	14.7	1.41	.07	5	14.7	1.4	.08
		Brown bullhead	5	10.6	0.6	.07	5	11.7	0.9	.05
		White perch	5	9.8	0.6	.21	5	9.4	0.5	.22
		White perch (R)	5	9.7	0.6	.17				
						Avg. .11				Avg. .12
#5	Susquehanna River Conowingo Dam, Md.	Carp	3	21.6	5.0	.07	4	20.0	3.5	.05
		Channel catfish	4	12.1	0.6	.05	5	13.6	0.9	.06
		Yellow perch	5	8.8	0.3	.09	5	9.1	0.4	.09
		Yellow perch (R)	5	7.9	0.2	.10				
						Avg. .07				Avg. .07
#6	Potomac River Little Falls, Md.	Carp	3	13.8	1.3	.08	5	15.1	1.9	.10
		Carp (R)	3	14.1	1.1	.05				
		White sucker	2	11.5	0.7	<.05	5	12.7	0.8	.08
		Black crappie	5	8.7	0.3	.09				
		Largemouth bass					5	10.5	0.5	.09
						Avg. .05				Avg. .09
#55	James River Richmond, Va.	Redhorse sucker	5	10.8	0.7	.11				
		Channel catfish	4	18.8	2.5	.12				
		Channel catfish (R)	3	17.3	2.5	.20				
		Largemouth bass	5	10.0	0.7	.14				
						Avg. .14				
#7	Roanoke River Roanoke Rapids, N. C.	Redhorse sucker	3	21.7	3.7	.08	5	19.0	2.8	.10
		Redhorse sucker (R)	3	20.0	3.2	.12				
		Brown bullhead	5	9.6	0.3	.21	5	9.6	0.4	.13
		Largemouth bass	3	12.0	1.0	.12	4	9.7	0.6	.09
						Avg. .14				Avg. .11
#8	Cape Fear River Elizabethtown, N. C.	Gizzard shad	5	6.2	0.2	.25	5	12.0	0.6	.18
		Channel catfish					2	21.5	3.9	.35
		Brown bullhead	5	8.2	0.3	.25	5	10.8	0.6	.17
		Brown bullhead (R)	5	8.2	0.3	.25				
		Largemouth bass	5	13.2	1.3	.60				
						Avg. .37				Avg. .23
#56	Pee Dee River Dongola, S. C.	White catfish	5	7.4	0.2	.19				
		White catfish (R)	5	6.6	0.1	.20				
		Bluegill	5	5.2	0.1	.20				
		Largemouth bass	5	12.5	1.5	1.00				
						Avg. .47				
#9	Cooper River Summerton, S. C.	Carp	2	17.5	3.5	.05				
		Spotted sucker					5	12.6	1.2	.13
		Bluegill	5	5.6	0.2	.20	5	6.8	0.2	.09
		Largemouth bass	4	11.3	0.6	.20	5	13.4	1.3	.14
		Largemouth bass (R)	4	12.0	0.7	.24				
						Avg. .16				Avg. .12
#10	Savannah River Savannah, Ga.	Carp	2	26.0	6.9	.17	4	15.8	1.8	.36
		Bluegill	4	8.3	0.5	.56	3	7.3	0.3	.45
		Largemouth bass	3	11.3	1.0	1.80	4	10.5	0.8	1.00
		Largemouth bass (R)	3	9.0	0.5	1.40				
						Avg. .78				Avg. .60
#57	Altamaha River Doctortown, Ga.	Spotted sucker	5	15.2	1.4	.25				
		Spotted sucker (R)	5	11.2	0.7	.11				
		Bluegill	5	8.2	0.4	.15				
		Largemouth bass	5	12.2	1.3	.51				
						Avg. .28				
#11	St. Johns River Welaka, Fla.	Striped mullet	3	16.3	1.7	.07				
		Channel catfish	5	9.0	0.3	.06	5	11.0	0.9	<.05
		Channel catfish (R)	5	8.0	0.2	<.05				
		Redbreast sunfish					4	5.8	0.2	.07
		Largemouth bass	2	12.5	1.3	.43	3	17.3	3.2	.08
						Avg. .18				Avg. .05

TABLE 1.—*Mercury residues in fish, 1969 and 1970*—Continued

Station Number and Location		Species	1970 No. of Fish	1970 Average Size Length (Inches)	1970 Average Size Weight (Lb)	1970 Total Mercury (PPM)[1]	1969 No. of Fish	1969 Average Size Length (Inches)	1969 Average Size Weight (Lb)	1969 Total Mercury (PPM)[1]
ATLANTIC COASTAL STREAMS—Continued										
#12	St. Lucie Canal Indiantown, Fla.	Channel catfish	2	18.5	2.2	.27	1	27.0	10.0	.23
		Channel catfish (R)	2	18.0	2.3	.15				
		Bluegill	5	5.6	0.2	.06	5	7.6	0.4	.13
		Largemouth bass	5	11.2	0.9	.28	5	13.4	1.6	.34
						Avg. .18				Avg. .23
GULF COASTAL STREAMS										
#58	Suwanee River Old Town, Fla.	Spotted sucker	4	15.8	1.7	.13				
		Redbreast sunfish	4	7.0	0.5	.13				
		Redbreast sunfish (R)	4	7.0	0.5	.21				
		Largemouth bass	5	9.0	0.4	.37				
						Avg. .22				
#13	Apalachicola River Jim Woodruff, Fla.	Spotted sucker	3	17.3	2.5	.10	5	17.4	2.3	.23
		Channel catfish	5	11.0	0.7	.11	5	11.6	0.9	.08
		Largemouth bass	5	10.8	0.7	.13				
		Largemouth bass (R)	5	9.4	0.5	.10				
						Avg. .11				Avg. .16
#59	Alabama River Chrysler, Ala.	Striped mullet	4	15.0	1.3	<.05				
		Striped mullet (R)	4	15.0	1.2	<.05				
		Bluegill	5	6.6	0.3	.48				
		Largemouth bass	3	17.3	2.6	.60				
						Avg. .36				
#14	Tombigbee River McIntosh, Ala.	Carp	5	17.0	3.7	.15	5	20.8	4.6	.36
		Striped mullet	4	15.8	1.6	.17	5	16.0	1.8	.36
		Striped mullet (R)	4	15.8	1.6	.35				
		Largemouth bass	5	13.0	1.2	.92	5	14.0	1.5	.65
						Avg. .44				Avg. .46
#15	Mississippi River Luling, La.	Carp	4	15.3	2.1	<.05	5	13.4	1.7	.11
		Carp (R)	4	15.3	2.4	.05				
		Freshwater drum	3	12.0	0.7	.30				
		Striped mullet					4	14.3	1.1	.14
		Channel catfish	5	13.8	1.0	.10	5	13.0	0.9	.22
						Avg. .14				Avg. .16
#60	Brazos River Richmond, Tex.	Smallmouth buffalo	3	17.2	3.2	.06				
		Channel catfish	3	20.9	3.4	.10				
		Channel catfish (R)	3	17.1	1.8	.08				
		Longnose gar	5	25.1	1.5	.24				
						Avg. .13				
#61	Colorado River Wharton, Tex.	River carpsucker	3	13.3	1.5	.15				
		River carpsucker (R)	3	12.6	1.5	.19				
		Channel catfish	3	13.7	1.2	.18				
		Spotted bass	3	8.4	0.5	.14				
						Avg. .16				
#62	Nueces River Mathis, Tex.	Gizzard shad	5	11.4	0.5	<.05				
		Gizzard shad (R)	5	10.0	0.4	.06				
		Channel catfish	4	12.9	0.7	.06				
		Largemouth bass	5	10.9	0.9	.40				
						Avg. .17				
#16	Rio Grande Brownsville, Tex.	Gizzard shad	5	10.7	0.4	.08	5	11.4	0.6	<.05
		Gizzard shad (R)	5	12.1	0.7	.12				
		Channel catfish	2	16.8	1.5	.17	5	15.2	0.9	.06
		Blue catfish	2	16.2	1.3	<.05	5	13.5	0.7	.06
						Avg. .09				Avg. .04
#63	Rio Grande Elephant Butte Reservoir, N. Mex.	Channel catfish	2	23.9	5.9	.35				
		Bluegill	4	4.6	0.1	.10				
		Bluegill (R)	3	4.7	0.1	.13				
		Largemouth bass	2	18.6	4.5	.52				
						Avg. .33				

TABLE 1.—*Mercury residues in fish, 1969 and 1970*—Continued

Station Number and Location		Species	1970 No. of Fish	1970 Average Size Length (Inches)	1970 Average Size Weight (Lb)	1970 Total Mercury (PPM)[1]	1969 No. of Fish	1969 Average Size Length (Inches)	1969 Average Size Weight (Lb)	1969 Total Mercury (PPM)[1]
GULF COASTAL STREAMS—Continued										
#64	Rio Grande Alamosa, Colo.	Carp	5	17.2	2.4	<.05				
		White sucker	5	12.7	0.9	.16				
		White sucker (R)	5	13.4	1.0	.28				
		Brown trout	3	10.8	0.5	.18				
						Avg. .13				
#65	Pecos River Red Bluff Lake, Tex.	Gizzard shad	5	12.3	0.8	.05				
		Gizzard shad (R)	5	12.4	0.8	.05				
		Channel catfish	5	15.8	1.4	.42				
		Largemouth bass	5	10.6	0.7	.06				
						Avg. .18				
GREAT LAKES DRAINAGE										
#17	Genessee River Scottsville, N. Y.	White sucker	4	14.0	1.2	.15	5	15.1	1.5	.13
		Redhorse sucker (R)	4	13.2	0.9	.19				
		Rock bass	4	8.1	0.5	.39	5	7.2	0.3	.22
		Walleye					2	17.2	1.6	.25
		Northern pike	4	13.7	0.7	.17				
						Avg. .24				Avg. .20
#66	St. Lawrence River Massena, N. Y.	White sucker	3	17.2	1.5	.22				
		Yellow perch	5	7.0	0.2	.20				
		Yellow perch (R)	5	8.3	0.3	.18				
		Northern pike	4	20.6	2.1	.39				
						Avg. .27				
#18	Lake Ontario Port Ontario, N. Y.	Yellow perch	5	8.5	0.4	.86	4	10.4	0.6	.48
		Yellow perch (R)	5	8.4	0.4	1.00				
		White perch	5	8.3	0.4	1.30	5	9.5	0.5	.43
		Rock bass	5	6.5	0.2	.30	3	8.6	0.6	.65
						Avg. .84				Avg. .52
#19	Lake Erie Erie, Pa.	White sucker	5	18.0	2.7	.31	3	14.8	1.5	.10
		Freshwater drum	5	14.1	1.3	.43	5	13.5	1.1	.15
		Yellow perch	5	9.7	0.4	.23	5	9.4	0.4	.13
		Yellow perch (R)	5	8.9	0.3	.15				
						Avg. .31				Avg. .13
#20	Lake Huron Bay Port, Mich.	Carp	5	19.6	4.0	.07	5	16.3	2.1	<.05
		Channel catfish	5	16.2	1.4	.07	5	15.9	1.5	.13
		Yellow perch	5	9.1	0.3	.08	5	9.9	0.5	.09
		Yellow perch (R)	5	9.1	0.3	.05				
						Avg. .07				Avg. .07
#21	Lake Michigan Sheboygan, Wis.	Bloater	5	11.2	0.6	.09	5	12.0	0.8	.09
		Bloater (R)	5	9.4	1.9	.10				
		Yellow perch	5	11.0	0.6	.07	5	10.3	0.6	.27
						Avg. .09				Avg. .18
#22	Lake Superior Bayfield, Wis.	Bloater	5	10.3	0.3	.15	5	11.2	0.4	.16
		Lake whitefish	5	17.6	1.7	.08	5	16.1	1.2	<.05
		Lake whitefish (R)	5	18.1	1.9	.06				
		Lake trout	5	23.4	4.0	.29	4	22.0	3.0	.14
						Avg. .17				Avg. .10
MISSISSIPPI RIVER SYSTEM										
#67	Allegheny River Natrona, Pa.	Carp	5	14.8	2.0	.11				
		Carp (R)	5	14.8	2.0	.05				
		Bluegill	5	5.5	0.2	.11				
		Walleye	5	12.7	0.6	.18				
						Avg. .12				
#23	Kanawha River Winfield, W. Va.	Carp	5	8.9	0.4	.08	4	8.6	0.4	<.05
		Brown bullhead	4	6.4	0.2	<.05	4	11.9	0.8	<.05
		Brown bullhead (R)	4	6.5	0.2	<.05				
		White crappie	5	6.8	0.2	.11	5	7.6	0.2	<.05
						Avg. .06				Avg. <.05

TABLE 1.—*Mercury residues in fish, 1969 and 1970*—Continued

Station Number and Location		Species	1970 No. of Fish	1970 Average Size Length (Inches)	1970 Average Size Weight (Lb)	1970 Total Mercury (PPM)[1]	1969 No. of Fish	1969 Average Size Length (Inches)	1969 Average Size Weight (Lb)	1969 Total Mercury (PPM)[1]
MISSISSIPPI RIVER SYSTEM—Continued										
#68	Wabash River New Harmony, Ind.	Carp	5	16.8	2.3	.25				
		Channel catfish	3	12.4	0.7	.15				
		White crappie	5	8.6	0.3	.16				
		White crappie (R)	5	7.6	0.2	.15				
						Avg. .19				
#24	Ohio River Marietta, Ohio	Carp	2	15.1	2.4	.24	4	10.1	1.6	.22
		Redhorse sucker	4	11.7	0.6	.07	10	7.9	0.04	.20
		Channel catfish	4	14.8	0.8	.83	1	15.7	1.3	.39
		Channel catfish (R)	4	14.9	0.9	.68				
		Largemouth bass					5	11.3	0.7	.50
						Avg. .36				Avg. .33
#69	Ohio River Cincinnati, Ohio	Carp	3	18.5	3.1	.20				
		Carp (R)	3	18.6	3.3	.09				
		White crappie	5	8.6	0.3	.15				
		Sauger	2	13.9	0.9	.30				
						Avg. .20				
#70	Ohio River Metropolis, Ill.	Carp	5	15.0	1.8	.14				
		Channel catfish	5	11.5	0.5	.11				
		White crappie	5	9.9	0.5	.43				
		White crappie (R)	5	10.0	0.4	.37				
						Avg. .22				
#25	Cumberland River Clarksville, Tenn.	Carp	4	13.5	1.4	.11	5	11.8	0.8	.09
		Bluegill	5	5.8	0.1	.05	5	6.2	0.1	.11
		Bluegill (R)	5	5.8	0.1	.05				
		Largemouth bass	2	11.0	0.8	.15	5	11.8	0.8	.27
						Avg. .10				Avg. .16
#71	Tennessee River Savannah, Tenn.	Carp	5	16.0	1.9	.40				
		Channel catfish	4	12.8	0.6	.28				
		Channel catfish (R)	4	13.0	0.6	.27				
		Largemouth bass	5	15.2	2.2	.67				
						Avg. .45				
#72	Wisconsin River Woodman, Wis.	Carp	3	18.2	3.3	.37				
		Carp (R)	2	19.3	4.0	.42				
		Channel catfish	3	16.7	2.1	.11				
		Smallmouth bass	3	11.7	1.0	.60				
						Avg. .37				
#73	Des Moines River Keosauqua, Iowa	Carp	5	12.8	1.3	.07				
		Carp (R)	5	12.1	0.9	.11				
		Channel catfish	2	10.3	0.4	.07				
		Walleye	3	12.6	0.9	.24				
						Avg. .17				
#26	Illinois River Beardstown, Ill.	Carp	5	14.2	1.4	.10	5	15.3	1.9	.09
		Carp (R)	5	16.3	2.1	.10				
		Bigmouth buffalo	3	18.6	3.9	.08	5	16.7	2.7	.07
		White crappie	5	7.3	0.2	.21	5	8.9	0.4	.13
						Avg. .13				Avg. .10
#74	Mississippi River Little Falls, Minn.	White sucker	4	13.5	1.2	.21				
		White sucker (R)	4	13.8	1.2	.55				
		Yellow bullhead	2	10.2	0.8	.68				
		Northern pike	5	14.3	0.8	.37				
						Avg. .48				
#27	Mississippi River Gutenberg, Iowa	Carp	4	18.8	2.9	.11	5	13.9	1.4	.10
		Bluegill	4	8.3	0.4	.20	5	7.1	0.4	.13
		Bluegill (R)	4	8.0	0.4	.12				
		Largemouth bass	3	15.3	2.1	.33	5	12.0	1.0	.25
						Avg. .20				Avg. .16
#75	Mississippi River Cape Girardeau, Mo.	Carp	3	18.2	3.0	.20				
		Channel catfish	5	15.4	1.3	.11				
		Channel catfish (R)	5	15.7	1.2	<.05				
		White crappie	3	10.7	0.7	.15				
						Avg. .14				

TABLE 1.—*Mercury residues in fish, 1969 and 1970*—Continued

Station Number and Location		Species	1970				1969			
			No. of Fish	Average Size		Total Mercury (PPM)[1]	No. of Fish	Average Size		Total Mercury (PPM)[1]
				Length (Inches)	Weight (Lb)			Length (Inches)	Weight (Lb)	
MISSISSIPPI RIVER SYSTEM—Continued										
#76	Mississippi River Memphis, Tenn.	Carp	2	19.0	4.0	.17				
		Carpsucker	2	16.5	2.4	.46				
		Carpsucker (R)	2	17.0	2.9	.14				
		Freshwater drum	2	20.0	3.9	.15				
						Avg. .21				
#28	Arkansas River Pine Bluff, Ark.	Carp	2	25.0	8.9	.09	3	21.0	3.8	.14
		Smallmouth buffalo	2	16.5	2.9	.11	4	16.3	2.5	.08
		Smallmouth buffalo (R)	2	19.5	4.8	.13				
		Flathead catfish	2	23.5	5.5	.22	2	21.0	4.6	.15
						Avg. .14				Avg. .12
#29	Arkansas River Keystone Reservoir, Okla.	Carp	5	13.4	1.2	.06	5	14.5	1.5	.08
		Carp (R)	5	13.9	1.3	.10				
		Bluegill	4	5.4	0.1	.13	5	6.2	0.2	<.05
		Largemouth bass	3	14.3	1.7	.22	5	15.0	2.5	.14
						Avg. .14				Avg. .07
#77	Arkansas River John Martin Reservoir, Colo.	Carp	5	14.7	1.4	.05				
		Carp (R)	5	12.9	1.1	.06				
		Channel catfish	3	9.2	0.3	.05				
		Black bullhead	3	8.5	0.4	.07				
						Avg. .06				
#78	Verdegris River Oologah, Okla.	Carp	5	14.8	1.6	.13				
		Carp (R)	5	15.0	1.7	.05				
		Bluegill	5	4.7	0.1	.07				
		Largemouth bass	3	14.3	2.3	.44				
						Avg. .20				
#79	Canadian River Eufaula, Okla.	Carp	5	13.6	1.1	.06				
		Carp (R)	5	13.2	1.2	.21				
		Bluegill	4	5.5	0.1	.07				
		Largemouth bass	2	11.0	0.8	.10				
						Avg. .10				
#30	White River DeValls Bluff, Ark.	Carp	2	18.0	2.9	.14	1	24.0	7.5	.27
		Bigmouth buffalo	2	17.0	2.6	.35	3	15.7	2.0	.22
		Bigmouth buffalo (R)	2	21.0	4.8	.25				
		Channel catfish	2	15.5	1.4	.12	4	14.5	0.8	.13
						Avg. .19				Avg. .21
#80	Yazoo River Redwood, Miss.	Carp	2	20.0	4.5	.19				
		Smallmouth buffalo	2	16.5	3.0	.16				
		Smallmouth buffalo (R)	2	15.5	2.3	.10				
		Gizzard shad	5	5.8	0.1	.15				
						Avg. .16				
#81	Red River Alexandria, La.	Smallmouth buffalo	2	21.0	5.8	.10				
		Smallmouth buffalo (R)	2	20.0	5.2	.10				
		Freshwater drum	3	13.7	1.1	.24				
		White catfish	3	13.0	0.6	.21				
						Avg. .18				
#82	Red River Lake Texoma, Okla.	Carp	5	20.5	4.5	.13				
		Carp (R)	5	20.2	4.3	.14				
		Bluegill	4	6.6	0.4	<.05				
		Largemouth bass	4	13.2	0.9	.15				
						Avg. .10				
#83	Missouri River Hermann, Mo.	Carp	2	19.4	3.7	<.05				
		Carp (R)	2	18.8	2.9	.08				
		Bigmouth buffalo	2	17.9	3.7	.14				
		Channel catfish	3	18.3	2.0	.15				
						Avg. .11				
#31	Missouri River Nebraska City, Nebr.	Carp	3	17.1	2.4	.06	5	13.6	1.2	<.05
		Carp (R)	3	16.9	2.2	.07				
		Goldeye	4	13.0	0.9	.20	5	12.5	0.8	.07
		Channel catfish					5	13.0	0.8	<.05
						Avg. .14				Avg. <.05

TABLE 1.—*Mercury residues in fish, 1969 and 1970*—Continued

Station Number and Location		Species	1970 No. of Fish	1970 Average Size Length (Inches)	1970 Average Size Weight (Lb)	1970 Total Mercury (PPM)[1]	1969 No. of Fish	1969 Average Size Length (Inches)	1969 Average Size Weight (Lb)	1969 Total Mercury (PPM)[1]
MISSISSIPPI RIVER SYSTEM—Continued										
#32	Missouri River Garrison Dam, N. Dak.	Carp	3	17.2	2.2	.11	2	15.2	1.6	.14
		Goldeye	5	12.4	0.5	.24	5	10.8	0.3	.13
		Goldeye (R)	5	11.9	0.5	.17				
		Walleye	3	15.8	1.3	.20	4	17.6	1.4	.30
						Avg. .17				Avg. .19
#33	Missouri River Great Falls, Mont.	Goldeye	5	11.2	0.5	.20	5	12.9	0.5	.14
		Goldeye (R)	5	11.2	0.5	.13				
		Redhorse sucker	5	15.9	1.7	.16	5	16.9	2.0	.13
		Sauger	5	12.5	0.7	.24				
						Avg. .19				Avg. .14
#84	Big Horn River Hardin, Mont.	Carp	5	15.5	2.4	.35				
		Goldeye	5	10.6	0.4	.23				
		Goldeye (R)	5	10.9	0.5	.25				
		Channel catfish	5	19.1	3.7	.18				
						Avg. .26				
#85	Yellowstone River Sidney, Mont.	Carpsucker	5	11.9	1.0	.11				
		Goldeye	5	11.3	0.5	.15				
		Goldeye (R)	5	10.3	0.4	.15				
		Channel catfish	5	9.1	0.4	.08				
						Avg. .11				
#86	James River Olivet, S. Dak.	Carp	3	15.8	1.0	.13				
		Carp (R)	3	15.3	0.9	.13				
		Goldeye	5	11.0	0.8	.22				
		Freshwater drum	4	9.0	0.3	.08				
						Avg. .14				
#87	North Platte River Lake McConaughy, Nebr.	Carp	3	15.8	1.2	.14				
		Carp (R)	3	15.9	1.2	.10				
		Channel catfish	3	16.5	1.0	.12				
		Walleye	5	16.2	1.8	.09				
		Rainbow trout	3	19.6	3.2	.11				
						Avg. .11				
#88	South Platte River Brule, Nebr.	Carp	5	5.6	0.1	.08				
		White sucker	5	12.0	0.7	.11				
		White sucker (R)	5	10.2	0.5	.15				
		Green sunfish	3	4.8	0.1	.10				
						Avg. .10				
#89	Platte River Louisville, Nebr.	Carp	3	19.7	3.7	.24				
		Carp (R)	3	12.2	0.8	.11				
		Channel catfish	5	12.6	0.6	.12				
		White crappie	3	7.6	0.3	.13				
						Avg. .14				
#90	Kansas River Bonner Springs, Kans.	Carp	5	14.8	1.9	.10				
		Carp (R)	5	15.8	2.8	.13				
		Gizzard shad	5	7.2	0.2	.08				
		Freshwater drum	5	9.9	0.7	.33				
						Avg. .18				
HUDSON BAY DRAINAGE										
#34	Red River Noyes, Minn.	Goldeye	5	12.3	0.6	.19				
		Goldeye (R)	5	12.4	0.7	.19				
		White sucker					2	16.4	2.1	.36
		Channel catfish	3	15.6	0.7	.13				
		Sauger	2	12.2	0.5	.60	3	13.7	0.9	.41
						Avg. .31				Avg. .39

TABLE 1.—*Mercury residues in fish, 1969 and 1970*—Continued

Station Number and Location		Species	1970 No. of Fish	1970 Average Size Length (Inches)	1970 Average Size Weight (Lb)	1970 Total Mercury (PPM)[1]	1969 No. of Fish	1969 Average Size Length (Inches)	1969 Average Size Weight (Lb)	1969 Total Mercury (PPM)[1]
COLORADO RIVER SYSTEM										
#35	Green River Vernal, Utah	Carp	5	10.8	0.6	.14	5	11.0	0.9	<.05
		Flannelmouth sucker	5	16.7	1.4	.17	3	19.2	2.6	.10
		Flannelmouth sucker (R)	5	16.6	1.5	.24				
		Channel catfish	5	9.2	0.3	.20				
		Black bullhead					3	5.4	0.2	<.05
						Avg. .18				Avg. <.05
#36	Colorado River Imperial Reservoir, Ariz.	Carp	5	16.4	3.9	<.05	5	16.9	2.5	<.05
		Carp (R)	4	14.8	2.5	<.05				
		Channel catfish	5	12.3	0.9	<.05	3	9.2	0.2	<.05
		Largemouth bass	5	14.5	0.9	.05	5	10.3	0.5	<.05
						Avg. <.05				Avg. <.05
#91	Colorado River Havasu Lake, Ariz.	Carp	5	14.1	1.4	<.05				
		Carp (R)	5	14.1	1.4	.08				
		Channel catfish	5	10.6	0.7	<.05				
		Largemouth bass	5	13.1	0.7	.08				
						Avg. .06				
#92	Colorado River Lake Mead, Nev.	Carp	4	15.8	2.8	.05				
		Carp (R)	3	16.1	3.0	.05				
		Largemouth bass	4	14.7	2.0	.09				
						Avg. .07				
#93	Colorado River Lake Powell, Ariz.	Carp	3	12.7	0.5	.27				
		Largemouth bass	4	13.9	1.2	.23				
		Largemouth bass (R)	4	13.0	0.9	.18				
		Rainbow trout	5	13.4	1.1	.10				
						Avg. .19				
#94	Gila River San Carlos Reservoir, Ariz.	Carp	6	14.5	1.0	.11				
		Bluegill	6	4.7	0.1	.13				
		Largemouth bass	3	13.1	1.2	.22				
						Avg. .15				
INTERIOR BASINS										
#37	Truckee River Fernley, Nev.	Carp	5	13.2	1.1	.21	5	14.6	1.5	.37
		Carp (R)	5	12.7	1.1	.23				
		Brown bullhead	5	10.1	0.6	.35	5	10.2	0.7	.22
		Largemouth bass	5	11.5	1.1	.65	5	12.3	1.1	.53
						Avg. .41				Avg. .37
#38	Utah Lake Provo, Utah	Carp	5	16.7	2.4	<.05	5	17.0	2.1	.06
		Black bullhead	5	10.2	0.6	.07	5	9.8	0.5	<.05
		Black bullhead (R)	5	9.9	0.6	.05				
		White bass	5	10.4	0.5	.06	5	10.2	0.5	.06
						Avg. .04				Avg. .05
#95	Bear River Preston, Idaho	Carp	3	18.6	3.8	.21				
		Largescale sucker	5	17.1	2.7	.34				
		Largescale sucker (R)	5	16.1	1.9	.37				
		Yellow perch	5	7.9	0.3	.13				
						Avg. .23				
CALIFORNIA STREAMS										
#39	Sacramento River Sacramento, Calif.	Carp	5	10.9	0.8	.18	5	12.9	0.9	.11
		Carp (R)	5	12.3	1.2	.18				
		White catfish	5	8.3	0.3	.20	5	14.1	1.3	.19
		Largemouth bass	5	11.9	1.0	.22	3	10.8	0.6	.13
						Avg. .20				Avg. .14
#40	San Joaquin River Los Banos, Calif.	Carp	5	15.1	1.7	.23	5	14.7	1.3	.20
		Channel catfish	5	16.6	2.3	.16	5	16.1	1.2	.20
		Black crappie	5	11.6	1.2	.09	5	10.4	0.6	.14
		Black crappie (R)	5	10.8	1.1	.11				
						Avg. .16				Avg. .18

TABLE 1.—*Mercury residues in fish, 1969 and 1970*—Continued

Station Number and Location		Species	1970 No. of Fish	1970 Average Size Length (Inches)	1970 Average Size Weight (Lb)	1970 Total Mercury (PPM)[1]	1969 No. of Fish	1969 Average Size Length (Inches)	1969 Average Size Weight (Lb)	1969 Total Mercury (PPM)[1]
COLUMBIA RIVER SYSTEM										
#41	Snake River Hagerman, Idaho	Largescale sucker	5	13.0	1.0	.08	5	15.0	1.4	.08
		Largescale sucker (R)	5	12.6	0.9	.10				
		Rainbow trout	5	14.2	1.3	.18	5	13.2	1.0	.13
		Northern squawfish	3	15.4	1.7	.43	5	15.5	1.3	.37
						Avg. .23				Avg. .19
#42	Snake River Lewiston, Idaho	Carp	4	15.3	2.3	.25				
		Largescale sucker	5	15.8	1.8	.23	5	15.9	2.0	.10
		Largescale sucker (R)	5	15.3	1.6	.18				
		Smallmouth bass	5	11.0	0.8	.21	3	7.0	0.3	.15
		Northern squawfish					2	13.0	0.8	1.25
						Avg. .22				Avg. .50
#43	Salmon River Riggins, Idaho	Carp	4	13.1	1.5	.29				
		Largescale sucker	4	17.4	2.3	.42	5	15.2	1.6	.23
		Largescale sucker (R)	3	17.7	2.3	.39				
		Northern squawfish	2	13.1	1.0	1.70				
						Avg. .80				
#96	Snake River Ice Harbor Dam, Wash.	Bridgelip sucker	5	12.1	0.8	.12				
		Channel catfish	5	13.1	1.2	.90				
		Channel catfish (R)	5	14.3	1.3	.10				
		Northern squawfish	4	12.6	0.7	1.20				
						Avg. .61				
#44	Yakima River Granger, Wash.	Carp	5	11.2	1.1	.23				
		Carp (R)	5	11.3	1.0	.23				
		Black crappie					5	7.4	0.3	.13
		Largescale sucker	5	14.0	1.2	.07	5	13.3	0.9	.30
		Smallmouth bass	3	12.3	1.4	.27	2	10.0	0.9	.14
						Avg. .19				Avg. .19
#45	Willamette River Oregon City, Oreg.	Carp	3	19.0	5.0	.17				
		Largescale sucker	5	15.3	1.8	.37	5	13.9	1.2	.18
		Largescale sucker (R)	5	15.9	1.7	.33				
		Chiselmouth					4	10.2	0.5	.13
		White crappie					5	6.9	0.2	.23
						Avg. .26				Avg. .18
#46	Columbia River Bonneville Dam, Oreg.	Largescale sucker	5	16.9	2.0	.21	5	16.5	2.0	.27
		Chiselmouth					4	7.9	0.3	.15
		Northern squawfish	5	12.7	0.8	.67	5	13.1	1.0	1.25
		Northern squawfish (R)	5	13.8	1.1	1.10				
						Avg. .55				Avg. .56
#97	Columbia River Pasco, Wash.	Carp	5	12.6	1.3	.07				
		Largescale sucker	5	17.2	2.6	.16				
		Largescale sucker (R)	5	16.3	2.1	.14				
		Mountain whitefish	3	12.0	0.5	.19				
						Avg. .14				
#98	Columbia River Grand Coulee Dam, Wash.	Bridgelip sucker	5	14.1	1.2	.07				
		Walleye	5	14.3	1.1	.11				
		Walleye (R)	5	14.8	1.3	.12				
		Northern squawfish	5	12.6	0.9	.25				
						Avg. .15				
PACIFIC COASTAL STREAMS										
#47	Klamath River Hornbrook, Calif.	Klamath sucker	5	15.7	2.1	.27	5	13.5	1.2	.12
		Yellow perch	5	7.9	0.3	.20	5	8.9	0.4	.22
		Yellow perch (R)	5	8.2	0.3	.19				
		Largemouth bass	5	8.4	0.3	.12				
		Rainbow trout					5	11.6	0.8	.14
						Avg. .20				Avg. .16
#48	Rogue River Gold Ray Dam, Oreg.	Carp	5	15.2	2.6	.18				
		Bridgelip sucker					5	14.0	1.5	.16
		Brown bullhead	5	10.8	0.7	.47	5	9.5	0.5	.90
		Brown bullhead (R)	5	10.8	0.6	.51				
		Black crappie					4	7.0	0.3	.25
		Largemouth bass	2	9.5	0.6	.14				
						Avg. .27				Avg. .44

TABLE 1.—*Mercury residues in fish, 1969 and 1970*—Continued

Station Number and Location		Species	1970				1969			
			No. of Fish	Average Size: Length (Inches)	Average Size: Weight (Lb)	Total Mercury (PPM)[1]	No. of Fish	Average Size: Length (Inches)	Average Size: Weight (Lb)	Total Mercury (PPM)[1]
ALASKAN STREAMS										
#49	Chena River Fairbanks, Alaska	Longnose sucker	2	13.5	1.7	.05	5	15.0	1.2	.09
		Round whitefish	3	11.7	1.0	.06	5	10.0	0.2	.06
		Arctic grayling	3	10.2	0.6	.06	5	11.7	0.5	.06
						Avg. .06				Avg. .07
#50	Kenai River Soldatna, Alaska	Longnose sucker					5	15.5	1.5	.08
		Round whitefish	5	12.1	0.7	.10				
		Rainbow trout	5	16.3	1.5	.06	5	13.2	0.9	.06
		Lake trout	5	13.7	0.9	.26	5	14.6	0.9	.12
						Avg. .14				Avg. .09
HAWAIIAN STREAMS										
#99	Waikele Stream Waipahu, Hawaii	Tilapia [2]	2	6.1	0.1	.11				
		Cuban limia [3]	13	2.6	0.1	.06				
		Chinese catfish [4]	3	8.1	0.1	.43				
		Chinese catfish (R)	2	9.4	0.2	.22				
						Avg. .17				
#100	Manoa Stream Honolulu, Hawaii	Tilapia [2]	3	8.0	0.3	.09				
		Cuban limia [3]	6	3.5	0.1	.13				
		Chinese catfish [4]	3	10.3	0.3	.18				
		Chinese catfish (R)	3	8.8	0.2	.15				
						Avg. .13				

NOTE: (R) designates replicate field sample. To compute residue average, the replicate samples at a sampling station were averaged and this average used with the values for the other species at a station to compute the average; values less than the sensitivity level of 0.05 ppm were considered to be zero in computing averages.

[1] mg/kg—wet-weight basis—whole fish.

[2] *Tilapia mossambica.*

[3] *Limia vittata.*

[4] *Clarias fuscus* (Identification uncertain).

TABLE 2.—*Results of analyses for total mercury and methyl mercury by Laboratory C and confirmatory analyses for total mercury by Laboratory H—1970*

Station Number and Location		Species	Total Mercury (mg/kg)[1]		Methyl Mercury [2] (mg/kg of Hg)
			Laboratory C	Laboratory H	Laboratory C
#1	Stillwater River	Chain pickerel	.64		.69
#2	Connecticut River	White perch	.51	.54	.43
		Yellow perch	.30	.38	
#3	Hudson River	Goldfish	.07	.13	
#4	Delaware River	White perch	.21	.24	
		White perch (R)	.17	.22	
#8	Cape Fear River	Largemouth bass	.60		.61
		Gizzard shad	.25	.17	
#10	Savannah River	Largemouth bass	1.80	1.73	1.70
#14	Tombigbee River	Largemouth bass	.92	.80	1.10
#15	Mississippi River	Carp (R)	<.05	.04	
#16	Rio Grande	Channel catfish	.17	.15	
#18	Lake Ontario	White perch	1.30	.97	1.00
#20	Lake Huron	Carp	.07	.04	
#21	Lake Michigan	Bloater (R)	.07	.09	

TABLE 2.—*Results of analyses for total mercury and methyl mercury by Laboratory C and confirmatory analyses for total mercury by Laboratory H—1970*—Continued

Station Number and Location	Species	Total Mercury (mg/kg)[1]		Methyl Mercury [2] (mg/kg of Hg)
		Laboratory C	Laboratory H	Laboratory C
#23 Kanawha River	Brown bullhead (R)	<.05	.03	
#24 Ohio River	Channel catfish	.83	.63	.65
	Channel catfish (R)	.68	.57	.56
#26 Illinois River	White crappie	.21	.12	
#28 Arkansas River	Smallmouth buffalo (R)	.13	.16	
#30 White River	Bigmouth buffalo (R)	.25	.24	
#31 Mississippi River	Goldeye	.20	.18	
#34 Red River (North)	Sauger	.60		.48
#37 Truckee River	Largemouth bass	.65		.65
#39 Sacramento River	Carp (R)	.18	.20	
#40 San Joaquin River	Black crappie	.09	.08	
#43 Salmon River	Northern squawfish	1.70		1.40
#45 Willamette River	Largescale sucker (R)	.37	.45	.36
#46 Columbia River	Northern squawfish (R)	1.10	.97	.88
#48 Rogue River	Brown bullhead	.47		.39
#51 Kennebec River	Yellow perch	1.20		1.10
#52 Lake Champlain	Yellow perch (R)	.49		.48
#53 Merrimac River	Yellow perch	.33	.34	
#54 Raritan River	Golden shiner	.08	.06	
#55 James River	Channel catfish	.12	.07	
#56 Pee Dee River	Largemouth bass	1.00	1.00	1.00
#57 Altamaha River	Spotted sucker	.25	.23	
	Largemouth bass	.51		.52
#59 Alabama River	Largemouth bass	.60	.74	.60
#63 Rio Grande	Largemouth bass	.52		.52
#67 Allegheny River	Walleye	.28	.19	
#69 Ohio River	Carp (R)	.20	.10	
	White crappie	.15	.19	
#71 Tennessee River	Largemouth bass	.67		.69
#72 Wisconsin River	Smallmouth bass	.60		.56
#74 Mississippi River	Yellow bullhead	.68		.62
#75 Mississippi River	Carp	.20	.16	
#76 Mississippi River	Freshwater drum	.15	.23	
#80 Yazoo River	Carp	.19	.21	
	Smallmouth buffalo	.16	.15	
#81 Red River	Smallmouth buffalo (R)	.10	.06	
#83 Missouri River	Channel catfish	.15	.19	
#96 Snake River	Northern squawfish	1.20	1.13	.94

NOTE: (R) designates replicate sample.
[1] Confirmatory analyses at Laboratory H on 40 samples also analyzed at Laboratory C.
[2] Analyses for methyl mercury at Laboratory C based on 24 samples.

Dursban® and Diazinon Residues in Biota Following Treatment of Intertidal Plots on Cape Cod—1967-69

Vahe M. Marganian[1] and William J. Wall, Jr.[2]

ABSTRACT

The effects of the organophosphorous pesticides Dursban® and diazinon on intertidal biota were studied over a period of 3 years, 1967-69. One percent granular Dursban® applied manually at an optimum concentration of 0.05 lb/acre controlled Culicoides *larvae effectively with no noticeable harm to fiddler crabs or other organisms. Residues recovered ranged from trace amounts to 2.30 ppm in white oligochaete, 2.58 ppm in ribbed mussel, 4.62 ppm in fiddler crab, 14.0 ppm in horsefly, and 15.7 ppm in marsh snail. Two percent granular diazinon applied manually at 0.20 lb/acre controlled* Culicoides *effectively, but killed small sand organisms. In general, concentrations of diazinon residues recovered were higher than those for Dursban® in the same organisms reported above. This report describes the sampling and gas chromatographic analytical procedures employed in this work, discusses data collected on residues in organisms at various periods after treatment, and gives persistence periods for these pesticides in substrates of intertidal sand, salt marsh sod, salt marsh mud, and sea water.*

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Introduction

In recent years, the deleterious effects of organochlorine pesticides, particularly DDT, on intertidal biota have received much criticism from environmentalists and the public. In an effort to find alternate means to organochlorine pesticide control, the effects of a number of organophosphorous pesticides on the breeding patterns of bloodsucking insects in intertidal areas have been studied (*1*).

This paper reports the results of a 3-year study (1967-69) conducted on Cape Cod, Mass., to determine if the use of Dursban® and diazinon for control of larvae of *Culicoides melleus* breeding in intertidal sand, *C. hollensis* and *C. furens* breeding in salt marsh mud, and *Tabanus nigrovittatus* and *T. lineola* breeding in salt marsh sod, would result in harmful effects to nontarget organisms and to determine residue levels in the intertidal biota. Reports on the field tests and on the classification of organisms are being published separately.

Because techniques for the chemical analysis of Dursban® and diazinon were not readily available, it was necessary to develop analytical methodology in the early

phases of the study for the detection and quantitative measurement of both compounds.

Treatment and Sampling Procedures

The majority of the tests were conducted in small intertidal sand and salt marsh plots ranging from 0.08 to 0.25 acres in size. For each test, two plots were treated with a single concentration of Dursban® or diazinon and a nearby untreated plot was used as a control. These pesticides, in granular form, were applied by hand at low tide, and in some instances uncoated granules were added to the pesticide-coated granules to provide additional materials for better distribution. During the latter phases of the program, two extended areas of intertidal sand or beach areas were treated using a Kiekens Whirlwind gasoline-operated duster and a salt marsh was treated using a helicopter.

Samples of intertidal biota, sand, and water were collected at regular intervals from treated and untreated areas before and after treatment as described by Wall and Marganian (*1*). When it was necessary to separate small organisms from the underlying sand, mud, or sod, the methods used for separation and counting of the larvae were based on that of Jamnback and Wall (*2*), Wall and Jamnback (*3*), and Wall and Doane (*4*).

The procedure for sample storage was based on methodology recommended by Van Middelem (*5*), Beckman (*6*), and Lykken (*7*). All specimens were stored at low temperatures (about 0° C) to prevent possible decomposition of the insecticides, and, when possible, samples were analyzed within 24 hours of their arrival at the laboratory.

Analytical Procedures

Extraction and cleanup procedures were related to techniques outlined by Thornburg (*8*) and Morley and Chiba (*9*). The wet weight of analyzed samples, representing composites of several specimens of the same species, as a rule, was in the following ranges: 1-10 g for claims, fiddlers, ribbed mussels, oysters, and shrimp; 0.1-1 g for mud snails, marsh snails, mud whelks, and periwinkles; 0.01-0.1 g for amphipods, crabs, tanaidaceans, horseflies, white oligochaetes, and red oliogochaetes; 15-20 g for sand; 50-55 g for sod; and 50 ml for water. Each sample of biota, sand, mud, and water was treated in a blender with 25 ml of reagent grade acetonitrile (density = 0.78 g/ml) for 5 minutes, and a 10-μl solution of internal standard dissolved in petroleum ether was added to the blender to insure uniform losses of insecticides during subsequent extractions. For the quantitative analysis of Dursban®, diazinon was used as an internal standard, and vice versa.

This was followed by a second extraction of the sample with 15 ml of acetonitrile, and the two extracts were then combined. The combined extracts were next treated with an equal volume of petroleum ether (b.p. = 30°-60° C, density = 0.62 g/ml) by shaking the mixture vigorously in a separatory funnel and removing the acetonitrile phase. This step was repeated twice using 25 ml of petroleum ether in each treatment of the acetonitrile phase, and all three petroleum ether extracts were combined. Five milliliters of a saturated NaCl solution and 25 ml of distilled water were added to the combined petroleum ether phase, shaken again, and the ether phase was transferred to a second separatory funnel, where it was washed again with distilled water. The two aqueous layers were treated together with small amounts of additional petroleum ether, and all ether phases were combined and dried over anhydrous Na_2SO_4. The petroleum ether extracts were next concentrated to about 0.3 ml with a gentle stream of nitrogen, and 2-μl aliquots were subjected to gas chromatographic analysis.

An F&M series Model 400 Biomedical gas chromatograph with a Honeywell strip chart recorder and a modified flame detector was used. This modification was achieved by coating a 27-gauge platinum wire with Na_2SO_4 beads and mounting the coiled wire over the tip of the flame. The operating parameters were as follows:

Columns:	Glass, 6' x ¼", o.d., packed with 5% UC-W98 on 60/80 mesh, acid-washed Diatoport S.
Temperatures:	Detector 245° C Injector 250° C Oven 185° C
Flow rates:	Carrier gas—He at 45 ml/min Oxygen (air) at 340 ml/min Hydrogen at 22 ml/min
Retention times:	Diazinon 4.2 min Dursban® 7.8 min

The lower limit of sensitivity was 0.01 ppm (mg/kg, net fresh weight). Quantitative calculations were carried out on a computer. A 6% precision of results was obtained on several samples determined at random. Recovery rates for both insecticides ranged from 80-85%. Data in this report do not include a correction factor for percent recovery. A few samples containing high concentrations of Dursban® and diazinon were analyzed by ultraviolet spectroscopy (at 231 mμ and 246 mμ, respectively) and were found to give comparable results on gas chromatographic analysis.

Results and Discussion

The effects of applications of Dursban® and diazinon on nontarget intertidal biota are shown in Table 1. The applications listed in Table 1 effectively controlled *Culicoides* larvae. In addition, Dursban® and diazinon

TABLE 1.—*Effects of Dursban® and diazinon on intertidal biota after effective control of Culicoidae larvae*

Application Rate lb/acre	Means of Application	Area Treated	Organism Controlled	Effects on Nontarget Organisms
		DURSBAN®		
0.05	duster	0.5 mile of intertidal shoreline	*C. melleus* larvae (up to 2 weeks following application)	Killed numerous fiddler crabs
0.05	manually	Sand plots	*C. melleus* larvae	No visible effects
0.1 and 0.2	manually	Sand plots	*C. melleus* larvae	Killed numerous fiddler crabs
0.20	manually	0.08—acre salt marsh plots	*Culicoides* larvae, specifically *C. furens* and *C. hollensis* (for 18 days)	Killed numerous larvae (75%)
		DIAZINON		
0.20	helicopter	17 acres of salt marsh	*C. hollensis* larvae (up to 29 days after treatment)	Numerous fish were found dead in the tidal creek up to 3 days following treatment
0.2	duster	1.1 mile of intertidal sand	*C. melleus* larvae	Killed numerous marine fish including *Fundulus* spp.
0.2	manually	Sand plots	*C. melleus* larvae	Killed small sand organisms such as naidids and dolichopodids
0.20	manually	0.12—acre salt marsh plots	*Culicoides* larvae, specifically *C. furens* and *C. hollensis* (for 7 days)	Killed numerous larvae (80%)

applied by hand at low tide to 0.25-acre salt marsh plots at rates of 0.05 and 0.3 lb/acre, respectively, failed to effectively control larvae of *Tabanus* sp.; numerous confined *Fundulus* spp. appeared to have died on the second day following this Dursban® treatment, and others placed daily in potholes were killed up to 4 days following the diazinon treatment.

An optimum concentration of 0.05 lb/acre of 1% granular Dursban®, applied manually, effectively controlled *Culicoides* larvae in small plots without visibly harming fiddler crabs or other organisms. However, Dursban® applied at the same rate by duster to 0.5 mile of intertidal sand shoreline controlled *C. melleus* larvae up to 2 weeks following treatment but also killed numerous fiddler crabs.

Two percent granular diazinon applied manually at 0.20 lb/acre controlled all *Culicoides* larvae effectively, but killed small sand organisms, such as naidids and dolichopodids.

Table 2 gives the results of analyses of various species from sand and salt marsh plots for residues of Dursban® following treatment at 0.05 lb/acre. Results of analyses of biota following diazinon treatments at 0.2 and 0.25 lb/acre are given in Table 3.

Some specimens from most of the species or groups (where species could not be determined) analyzed during the first 4 days after treatment were found to contain measurable amounts of Dursban® (Table 2) or diazinon (Table 3). Organisms taken from sand plots treated with diazinon applied at the rate of 0.2 pound of technical material per acre generally contained more residues than similar organisms taken from comparable plots treated with Dursban® applied at the rate of 0.05 pound of technical material per acre. Table 4 shows the periods of persistence of these pesticides in intertidal sand, salt marsh sod, salt marsh mud, and sea water. Each figure in Tables 2-4 represents one analysis per sample.

With a few exceptions, no measurable residues of Dursban® or diazinon were found in the organisms from or the substrate of the treated or control plots prior to treatment, and none were found in organisms or substrate from control plots following treatment.

In most of the species or groups analyzed, no obvious pattern of accumulation or disappearance of residues could be established for the two pesticides studied. In some specimens, residues up to 50 ppm were found during the first few days following treatment, gradually declining to immeasurable amounts at the end of 30 days.

TABLE 2.—*Dursban® residues in marine organisms at various intervals after treatment at a concentration of 0.05 lb/acre*

Organism	Substrate	Dursban® Residues in PPM (fresh-weight basis)[1]								
		Days								
		1	2	3	4	7	9	10	12	22
Oligochaeta										
Enchytraeidae (white oligochaete)	Salt marsh mud	1.0	—	2.30	—					
Mya arenaria (clam)	Intertidal sand		T	—	—	0.01				
Mollusca										
Modiolus demissus (ribbed mussel)	Salt marsh mud	—	2.58	0.12			—			
Nassarius obsoletus (mud snail)	Intertidal sand	T	T	1.10	—	0.62				
Melampus bidentatus (marsh snail)	Salt marsh mud	—	T	2.24	—	—		15.70		
Arthropoda										
Leptochelia sp. (tanaidacean)	Salt marsh mud	T	—	16.70			—			T
Palaemonetes sp. (prawn)	Intertidal sand	—	—							
Uca spp. (fiddler crab)	Intertidal sand	T	T	—		4.62				
Culicoides spp. (gnat)	Salt marsh mud	0.43	—							
Tabanus spp. (horsefly)	Salt marsh mud	T	—	3.80		0.43		14.00	—	

NOTE: Dursban® applied as 1% technical grade Dursban® in granular form; blank = no sample taken; — = no residue detected; T = trace = <0.01 ppm.

[1] Represents one analysis per sample.

In other organisms of the same species or group, collected during the second or third week following treatment, residues over 20 ppm were found.

From 1 to 7 days following treatment, many annelids, particularly Oligochaeta, did not contain measurable amounts of residues, but others were found to contain large quantities (over 10 ppm) of the particular pesticide employed. From 1 to 21 days following treatment, samples of the *Fundulus* spp. (killifiish) as well as many samples of Pelecypoda and Arthropoda analyzed contained small measurable amounts of residues (usually <1 ppm) of the pesicide distributed. In general, Mollusca (Gastropoda and Pelecypoda) were found to consistently contain more residues than the other organisms analyzed.

Organisms taken from salt marsh habitats generally contained more residue of the pesticide employed than the same species or group taken from intertidal sand habitats.

Dursban® applied at the rate of 0.05 pound of technical material per acre persisted in measurable amounts in the top one-half inch of intertidal sand for an average of 2 days with a maximum of 4 days; in the top inch of salt marsh sod for an average of 5 days with a maximum of 12 days; and in the top inch of salt marsh mud for an average of 15 days with a maximum of 22 days (Table 4). Diazinon applied at the rate of 0.2 pound of technical material per acre persisted in measurable amounts in the top one-half inch of intertidal sand for an average of 4 days with a maximum of 12 days; in the top inch of marsh sod for an average of 6 days with a maximum of 10 days; and in the top inch of marsh mud for an average of 10 days with a maximum of 22 days (Table 4). For both pesticides, residues on the day following treatment were greatest, gradually declining thereafter.

With one minor exception, analysis of sea water taken from the bay or creek immediately adjacent to each treated plot showed no residues on the day following treatment.

During the summer following the original application of Dursban® in small intertidal sand and marsh plots, organisms and substrates from these plots were analyzed, and no measurable amounts of residues of either pesticide were found.

TABLE 3.—*Diazinon residues in marine organisms at various intervals following treatment*

Organism	Substrate	Application Rate lb/acre	Diazinon Residues in PPM (fresh-weight basis)[1]																
			Days																
			1	2	3	4	5	6	7	8	9	11	14	15	22	29	36	38	45
Oligochaeta																			
Enchytraeidae (white oligochaete)	Intertidal sand	0.20		14.82		—	—												
Naididae and *Tubificidae* (red oligochaete)	Salt marsh	0.25									100					36.80			
Polychaeta																			
(sandworm)	Salt marsh	0.25					0.73				7.82			23.51		—			
Mollusca																			
Mercenaria mercenaria (quahog)	Salt marsh	0.25	4.36	T		0.07	1.80												
Crassostrea virginica (oyster)	Salt marsh	0.25				0.56				1.69	0.81		—	0.11	0.03	3.16		—	—
Modiolus demissus (ribbed mussel)	Salt marsh	0.25	50.00	2.06		2.93	3.93		0.99			0.09	0.23			T	0.03		
Nassarius obsoletus (mud snail)	Intertidal sand	0.20	—	—	0.16		0.02		2.45				0.30	0.23				—	0.17
Melampus bidentatus (marsh snail)	Salt marsh mud	0.20	2.22	1.31		2.22			—			0.25	1.66			—			
Littorina littorea (periwinkle)	Intertidal sand	0.20		0.62		0.11	2.16	14.26	T		T			0.16		0.40			
Uca spp. (fiddler crab)	Salt marsh mud	0.20	0.05	—	—	0.33	T	—	0.11				0.04	T	0.01	—	—	—	0.09
Carcinas maenas (green crab)	Intertidal sand	0.20	0.82			—													
Pagurus longicarpus (hermit crab)	Salt marsh	0.25	3.61			0.46	1.21								0.12				
Palaemonetes and *Crangon* (prawn)	Salt marsh mud	0.20				0.86													
Pisces																			
Fundulus spp. (killifish)	Intertidal sand	0.20	1.42	0.95		0.95	0.10					0.49							

NOTE: Diazinon applied as 2% technical grade in granular form; blank = no sample taken; — = no residue detected; T = trace = <0.01 ppm.

[1] Represents one analysis per sample.

TABLE 4.—*Persistence of Dursban® and diazinon in various marine substrates*

Substrate	Days			
	Dursban		Diazinon	
	Average	Maximum [1]	Average	Maximum [1]
Intertidal sand	2	4	4	12
Salt marsh sod	5	12	6	10
Salt marsh mud	15	22	10	22
Sea water		0		0

[1] Time of last detectable trace.

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See Appendix for chemical names of compounds discussed in this paper.

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PESTICIDES IN WATER

Seasonal Variations in Residues of Chlorinated Hydrocarbon Pesticides in the Water of the Utah Lake Drainage System—1970 and 1971 [1]

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ABSTRACT

Definite surges of pesticides (1 ppb or more) enter Utah Lake three times per year—early spring, late spring, and fall, generally corresponding to the application times of pesticides by farmers in the area. The pesticides involved were mainly aldrin and BHC in the early spring; heptachlor (plus heptachlor epoxide) and methoxychlor in the late spring; and aldrin, heptachlor, and methoxychlor in the late fall. The fish samples collected from Utah Lake contained only small amounts of pesticides, the highest level being 956 ppb DDE.

Introduction

Utah Lake is a large freshwater lake in central Utah (Fig. 1), 25 miles long and 11 miles wide with an average depth of 9 feet. The major inlets to Utah Lake are on the east side, and more than half of the estimated 600,000 acre-feet of water entering Utah Lake in an average year enters through the Provo, Spanish Fork, and American Fork Rivers. The rest of the water enters through agricultural drains, small creeks, and underground springs. The only outlet from Utah Lake is the Jordan River to the north. Utah Lake serves as a reservoir for the water users of the Utah Lake and Jordon River Irrigation System.

Previous studies concerning the overall chemistry of Utah Lake were summarized by Bradshaw *et al.* (*1, 2*). The only previous pesticide residue studies of Utah Lake showed that at least two surges of pesticides enter Utah Lake every year (*1, 3*). Fish from Utah Lake were reported to contain minute amounts (0.1-0.8 ppm) of chlorinated hydrocarbon residues (*4, 5*). This study was undertaken to catalog the sources and types of pesticides entering the Lake in a given year and to determine the extent of pesticide residues in Utah Lake fish.

Sampling and Analytical Procedures

WATER SAMPLES

Water from 15 tributaries and 1 outlet point (16 Stations) was sampled biweekly from March 1 to July 1, 1970, and weekly or semiweekly through February 1971 (Fig. 1). Station 2 was soon abandoned because it dried up. One gallon of water was collected at each station in large mouth glass jars. The water (3 liters) was extracted as soon as possible (within 3 days) with nanograde petroleum ether (*Mallinkrodt Chemical Co.*) in a continuous liquid-liquid extractor for 24 hours. Care was taken to insure that a representative mixture of water and suspended material was extracted. The petroleum ether extract was dried over anhydrous sodium sulfate, filtered, and evaporated to 10 ml. The sample was then analyzed on a dual glass column, Varian Aerograph 202 gas-liquid chromatograph (GLC) using electron capture detectors. Each sample was analyzed simultaneously on the two columns. One column (⅛" x 5') was packed with a mixture of 4% SE-30 and 6% QF-1 on Chromosorb W and the other, with a mixture of 1.5% OV-17 and 1.95% QF-1 on Chromosorb W. The columns were heated to 200° C. The GLC peak areas were determined using a Disc Integrator. Quantitation was accomplished by comparing the peaks with

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those of known standards. Only pesticides verified by the second column were evaluated (*6*). The final pesticide levels were calculated using a Hewlett-Packard 9100 B "programable" calculator.

FISH SAMPLES

Fish were caught from the east side of Utah Lake between the Orem Pier and Provo Airport on July 21, 1970, by Utah State Wildlife Resources personnel. The fish were treated in a manner similar to that reported by Kleinert, Degurse, and Wirth (*7*). Each frozen fish sample was ground and homogenized in a heavy duty meat grinder. A 10-g portion of the homogenized fish was added to a blender and mixed with 60 g of anhydrous sodium sulfate and 200 ml of hexane (distilled through a glass column in the laboratory). The solvent from the blended material was decanted through a filter paper into a 500-ml round-bottom flask. The fish sample was washed four times with 50-ml portions of hexane. The combined hexane extracts were evaporated at about 50-mm pressure to 5 ml using a rotary evaporator. This material was then placed on a column containing 200 g of Florisil and eluted from the column with a mixture of 180 ml of hexane and 12 ml of ethyl ether which contained 2% ethyl alcohol. The solution was eluted at a rate of 5 ml/min. The resulting solution was evaporated at about 50-mm pressure to 10 ml and subjected to GLC analysis as reported above.

RECOVERY STUDIES

Spiked water samples treated as above gave 75-85% recovery of the various pesticide residues. The results were not corrected to reflect this recovery.

Results and Discussion

WATER SAMPLES

The concentrations of pesticides detected in water samples (aldrin, total alpha- and gamma-BHC, heptachlor and heptachlor epoxide, methoxychlor, and DDT-type compounds) by collection stations on Utah Lake are shown in Fig. 2. The results showed that definite surges of pesticides entered the Lake at specific times during 1970. Except for these surges, extremely low levels of pesticides were detected in the water entering the Lake. Aldrin and BHC were the main residues entering Utah Lake in the early spring (March-April); heptachlor or its epoxide and methoxychlor in the late spring (May-June); and aldrin, heptachlor or its epoxide, and methoxychlor in the late fall (October-November). These surges corresponded to the times of application of the respective pesticides in Utah County.

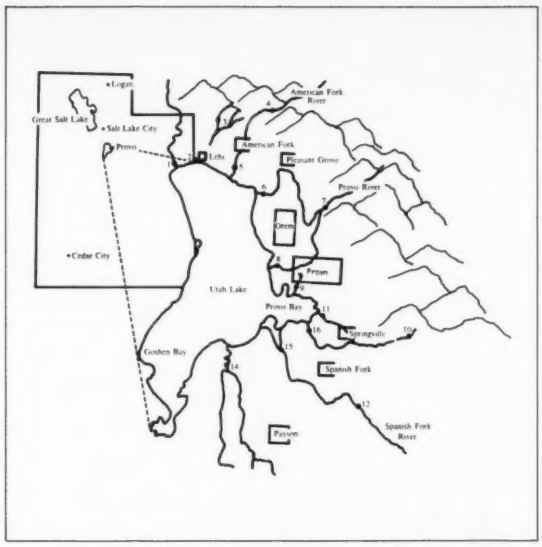


FIGURE 1.—*Sampling stations on the Utah Lake tributaries, 1970 and 1971*

Most of the pesticide residues were detected in tributaries in valley locations, with the exception of residues detected in the early spring (probably resulting from forest runoff) and at one location in the fall (high aldrin at Station 4). Station 13 (Fig. 1) was also considered a valley location since there are many farms above this point.

Aldrin (Fig. 2) was observed mainly in the northern part of the Utah Valley where it is used for grasshopper control. The spring and fall surges of this pesticide entering Utah Lake were at levels over 1 ppb.

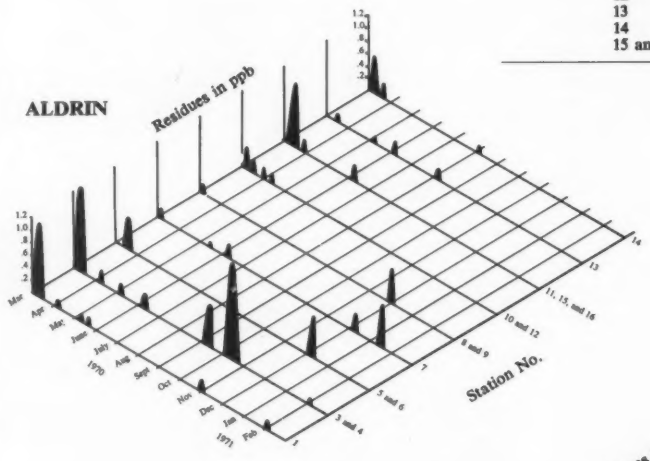
The greatest surge of BHC, which is used extensively on livestock in Utah County, was observed in the early spring at a level of 1.3 ppb (Fig. 2) in the south central area where there is much livestock.

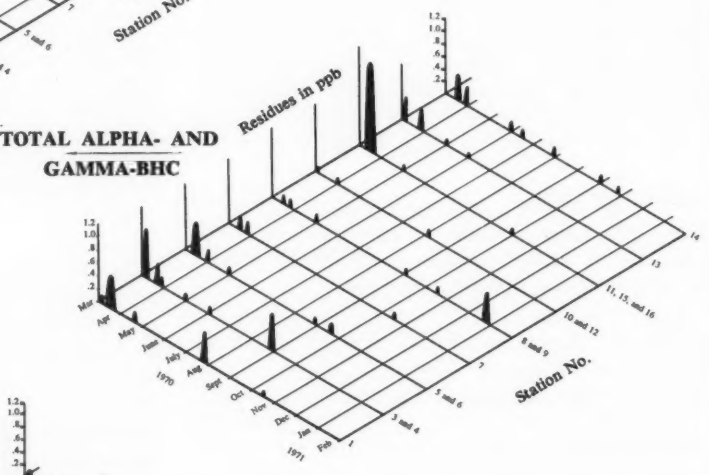
The high incidence of heptachlor or its epoxide in the alfalfa-producing area (Fig. 2—Stations 5, 6, and 13) was somewhat surprising, however, since heptachlor is no longer registered for use on alfalfa (*8*) and has not been sold in Utah County for 3-4 years. It is likely that the heptachlor used was that left over from previous purchases.

Methoxychlor, due to its relatively rapid degradation (*9*), is finding wide use as a replacement for DDT and was used extensively throughout Utah County. It was never detected in samples at the Jordan River outlet from Utah Lake probably because of its degradation (Fig. 2).

FIGURE 2.—*Pesticide residues in water samples from the Utah Lake drainage system, 1970 and 1971*

Sampling Station Number	Location
1	Jordan River
3 and 4	North Upper
5 and 6	North Lower
7	North Central Upper
8 and 9	North Central Lower
10	South Central Upper
11	South Central Lower
12	South Central Upper
13	South Upper
14	South Lower
15 and 16	South Central Lower





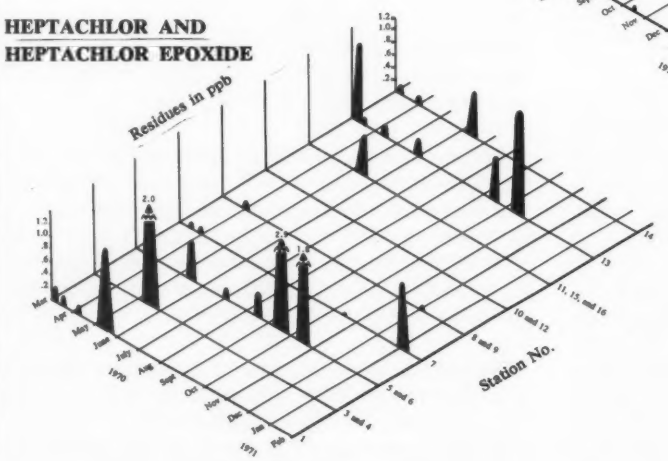
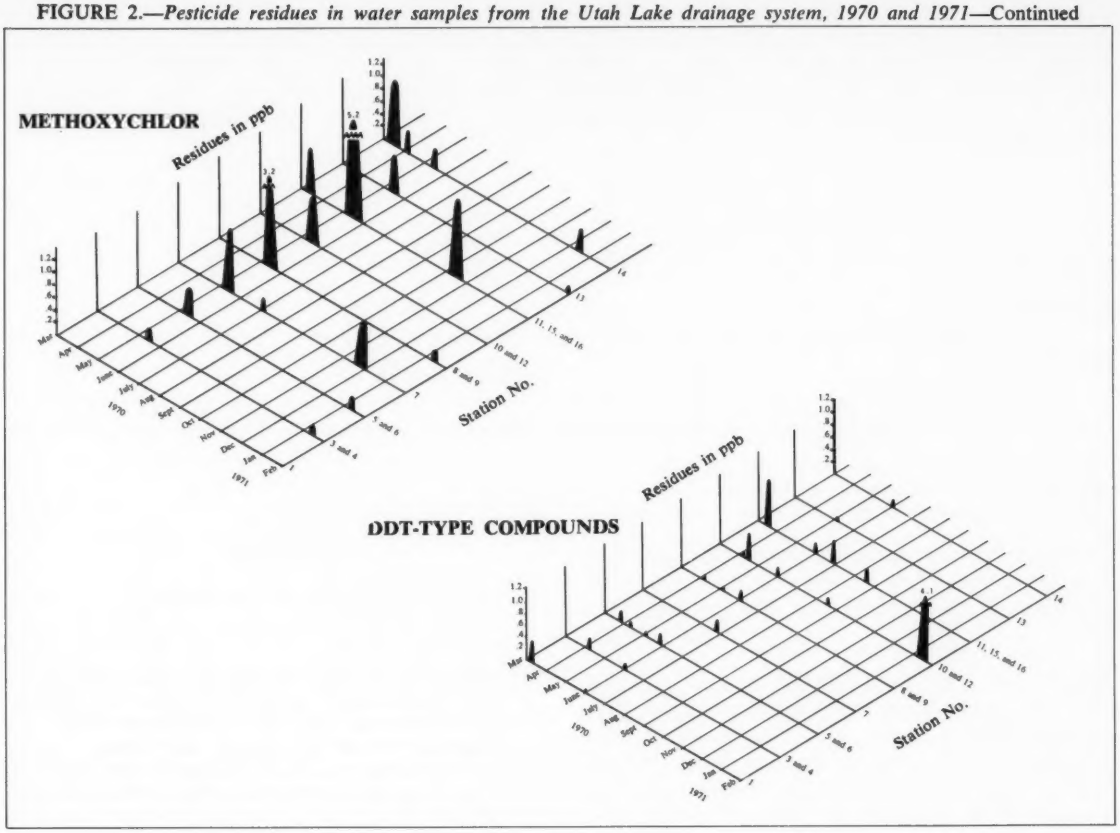


FIGURE 2.—*Pesticide residues in water samples from the Utah Lake drainage system, 1970 and 1971*—Continued



Except for isolated instances, DDE, the principal degradation product of DDT, was observed only at low concentrations in water samples (Fig. 2). The observed DDE could have been leached from the soil or washed into the waterway on soil particles. Indeed, every occurrence of DDE was preceded by a rain storm in the area (*11*). DDT was observed only once, at Station 12 in January 1971 at a high concentration (4.1 ppb). Very little DDT is presently being used in Utah Valley, and this high level may have been due to supplies of DDT having been dumped in Spanish Fork Canyon.

There are no known studies of the pesticide variations in the water of a small isolated basin such as the Utah Lake drainage system. There have been a number of national studies of pesticide residue levels in surface waters (*10*) in which one water sample from a large number of rivers was analyzed per year. The pesticides were found to be widespread throughout the country, but were below permissible limits as they relate to human intake directly from a domestic water supply (*10*).

FISH SAMPLES

Pesticide residue levels in the fish of Utah Lake have been reported previously (*4, 5*). In the study reported here, the most common pesticide observed in fish was DDE, probably due to its great persistence (Table 1). The smaller and younger fish contained lesser amounts of DDE.

It is evident that pesticide levels in fish were much higher than those found in water. Reinert (*12*) and Crosby and Tucker (*13*) have shown that accumulation of pesticides in fish results primarily from absorption from the water. Reinert (*12*) has shown that uptake of pesticides from water can be very rapid and that fish can attain up to 1 ppm of pesticide from water with concentrations as low as 1 ppt.

Although a limited number of fish samples were collected in this study, it is evident that the levels of pesticide residues in the fish in Utah Lake are very low when compared to those of fish in many other areas. Fish studied in the Midwest had from 2-10 ppm total pesti-

TABLE 1.—*Pesticide concentrations in Utah Lake fish, 1970*

FISH [1]	LENGTH (CM)	WEIGHT (G)	AGE [2] (YEARS)	PESTICIDES [3] (PPB)
Carp *(Cyprinus carpio)*	39.5	844	3	302 DDE, 230 Dieldrin
	43.5	1,044	4	288 DDE, 215 Dieldrin
	45.5	1,036	4	123 DDE, 30 BHC, 62 Methoxychlor
	44.5	1,135	5	369 DDE, 56 Methoxychlor
	49	1,667	7	956 DDE
Channel catfish *(Ictalurus punctatus)*	51	1,264	6	652 DDE, 79 DDT
Bullhead catfish *(Ictalurus melas)*	25.5	274	2	126 DDE
White bass [4] *(Morone chrysops)*	19	85	2	53 Dieldrin

[1] Fish were caught by Utah State Wildlife Resources personnel on the east side of Utah Lake between the Orem Pier and Provo Airport on July 21, 1970.

[2] Determined from scales and spine cross sections.

[3] Reported in parts per billion (ppb) of a whole fish.

[4] Four fish were ground together to provide a large enough sample.

cides (*4, 5*) as compared to 0.05-0.96 ppm in Utah Lake. The major reason for the low pesticide residues in Utah Lake fish might be the fact that relatively little farming is carried out in the area as compared to the Midwest. Another possible reason is that the pesticides are being adsorbed by the suspended silt particles in Utah Lake; however, this was not confirmed in this study:

Acknowledgment

The authors wish to thank those who participated in the project by collecting and extracting the water samples, in particular, E. M. Michenor, B. Hague, J. F. Brown, E. Loveless, and M. Morrison, and to thank the Utah State Agriculture Agents for their assistance in evaluating the data. We wish also to thank Joe White, Utah State Wildlife Resources, who provided the fish samples.

See Appendix for the chemical names of compounds discussed in this paper.

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Sampling Procedures and Problems in Determining Pesticide Residues in the Hydrologic Environment [1]

Herman R. Feltz[2] and James K. Culbertson[3]

ABSTRACT

Diligent use of standardized sampling and analytical techniques is essential to meaningful assessments of the occurrence, distribution, and fate of pesticide residues in the hydrologic environment. The validity of analytical data and subsequent interpretations are interdependent and limited to the confidence level of adequate, representative sampling of various components. Equally important are appropriate sample-preservation practices and procedures for sample preparation and cleanup and identification, measurement, and confirmation of residues. Analytical schedules should include pesticides listed in the Revised Chemicals Monitoring Guide for the National Pesticide Monitoring Program and should be responsive to special interests. Samplers are available for collecting acceptable water, fluvial material, and bottom-material samples in about 75% of the river miles and in lakes and estuaries throughout the United States; however, more experience is needed in sampling hydrosols and low density deposits at the active water-sediment interface.

Introduction

Scientific literature contains many reports pointing out the "pesticide problem" and presenting data used to assess the occurrence and distribution of pesticides in various components of the hydrologic environment. Most assessments have been thorough but limited to an individual component. Concentration levels in each aquatic phase must be related to the whole aquatic environment because each component exerts a control, creating an interdependence. To maintain a current assessment, data from all investigators have become increasingly important because these data are used to derive trends in residue concentrations and to identify possible problem areas.

Because measuring pesticide residues in the Nation's water resources is far too great for any single group, contributors are numerous and represent Federal, State, private, and academic organizations. Therefore, standardized sampling and analytical techniques must be used to acquire the valid and interrelated data needed for meaningful assessments of the occurrence, distribution, and fate of pesticide residues. Standardization should not be rigid or deter technical advancement, but there should be some minimum criteria established for sampling and analysis which investigators should fulfill.

Pesticides are distributed throughout the hydrologic environment—the atmosphere, precipitation, surface water, ground water, suspended and bed sediments, flora, and fauna. Measurements in each component are equally important, and validity begins immediately upon sampling. Irrespective of scrutiny and quality control applied in performing laboratory analyses, reported data are no better than the confidence that can be placed in the representativeness of the sampling. Webster's dictionary states that "a sample is a representative part from a whole whose properties are studied to gain information about the whole." Because the whole cannot be analyzed, the sample thus becomes paramount.

Although a high degree of analytical capability has generally been attained, the use of adequate samplers and sampling techniques has not been emphasized. Perhaps, the greatest weakness in assessing the "pesticide situation" is the lack of representative samples for analysis.

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[1] Presented to the Division of Pesticide Chemistry at the 161st National Meeting, American Chemical Society, Los Angeles, Calif., March 29, 1971.

[2] U.S. Geological Survey, Arlington, Va. 22209.

[3] U.S. Geological Survey, Washington, D.C. 20242.

This paper directs attention to the state of the art, the problems, and some of the pitfalls in collecting representative samples from streams, lakes, and estuaries.

Occurrence of Pesticide Residues

Pesticide residues have been detected in all components of the dynamic hydrosystem, attesting to the ubiquitous nature of these residues (*1, 10-13, 15*). The occurrence and distribution of residues are as divergent as the components. Concentrations range from the generally accepted lower detection limit of 0.01 μliter/liter in filtrates to 100,000 times this value in particulate matter separated from the same aqueous samples. The environment ranges from the most humid to the driest, and residues may originate from local applications or as fallout transported from remote areas.

In assessing the quality of water resources, the significance of measuring residues throughout the aquatic system is shown by the wide range in concentrations found within a single hydrologic area as illustrated in Table 1. This table, based on results of recent work in south Florida, indicates the order of magnitude of total DDT (DDT+DDE+DDD) residue values which ranged from 0.02 μg/liter in water up to 16,500 μg/kg in birds at the top of the food chain. The biological accumulation of DDT family residues in the ecosystem is several hundred thousand times the average concentration found in surface and ground water.

The least valid assessment of the distribution of pesticide residues is one based on data from the aqueous phase alone. Although water is primarily considered when studying the aquatic environment, evaluating pesticide residues is not truly significant without relating all components as an interdependent system.

Sampling Techniques

The data presented in Table 1 represent a cross section of an entire ecosystem in which each component presents sampling problems. The following discussion, however, emphasizes the identification of problems in sampling water, fluvial sediment, and bottom materials.

WATER AND FLUVIAL SEDIMENT

Preliminary results of an investigation of pesticide residues in runoff to streams draining different land-use areas in central Pennsylvania indicated that the concentration of the DDT family sometimes is directly correlated with suspended-sediment concentration (L. A. Reed and J. F. Truhlar, *personal communication, 1970*). Samples were collected using a depth-integrating sampler and the equal-transit-rate sampling procedure. Correlation curves for two of the streams in the study area are shown in Fig. 1.

TABLE 1.—*Scheme of biological accumulation of total DDT (DDT+DDD+DDE) in south Florida*[1]

Environmental Component	Concentration of Total DDT (DDT+DDD+DDE) in μg/liter for Water and μg/kg for Plants and Animals
Water:	
Surface	.02
Ground (in Biscayne Aquifer)	.02
Rain	.30
Everglades algal mats or periphyton (producers)	7
Everglades vascular plants (producers)	8
Everglades submerged soils	16
Everglades crustaceans (omnivores)	23
Everglades marsh fishes (omnivores and primary carnivores)	196
Everglades alligators (higher carnivores)	352
Everglades kite eggs and eagle (higher carnivores)	16,500

[1] Data from U.S. Geological Survey except eagle and kite data from National Park Service (Department of the Interior).

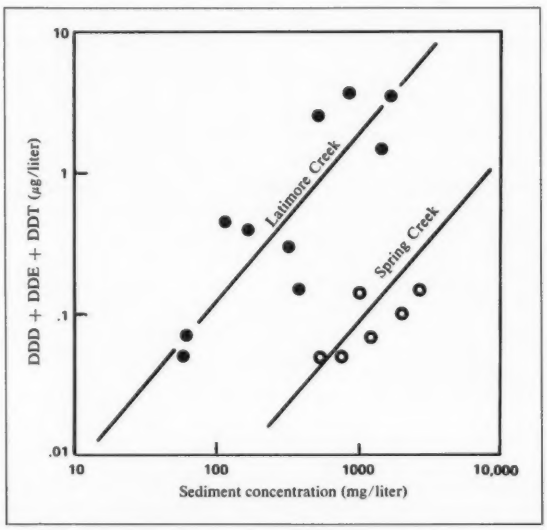

FIGURE 1.—*Correlation between suspended-sediment concentration and total concentration of DDD+DDE+DDT in unfiltered water samples*

More representative sampling of pesticide residues is based on a better understanding of the role of fluvial-sediment transport. Numerous reports reveal that residues associated with sediment are dependent upon both particle-size distribution and organic matter content (*12*). Therefore, the variability of pesticide concentrations may be correlated with the variability of suspended-sediment concentration in the cross section of a stream.

Water is usually sampled by filling a container held just beneath the surface of a body of water. Published data and discussions by investigators reveal that a high percentage of all samples have been obtained in this manner. This sample is commonly referred to as a dip sample. Using a weighted bottle holder which would allow the bottle to be lowered to any desired depth and returned to the surface would improve this method. If the person taking a sample could be assured that the bottle was lowered to the bottom and raised to the surface at a uniform rate, he would have roughly approached collection of what is known as a depth-intergrated sample.

A true depth-integrated sample is collected by means of a depth-integrating sampler which integrates discharge as a function of depth (*8, 18*). Sediment is maintained in suspension because of velocity and turbulence. Velocity varies from the water surface to the stream bed, being generally highest near the surface and lowest at the bed. Sediment concentration varies also from the surface to the bed, being lowest at the surface and greatest at the bed. Fine sediment (finer than about 0.062 mm) is easily kept in suspension and is distributed relatively uniformly throughout the depth of flow. However, as particle size increases, more energy is required to maintain suspension in a given flow, and the average size of sediment in suspension increases from the water surface to the bed. Depth integration is used to collect a water-sediment sample that is weighted according to velocity at each increment of depth. This means that the water-sediment mixture must enter the sample container at the same velocity as the flow passing the intake. If the depth-integrating sampler is lowered from the water surface to the bed and back at the same transit rate, each increment of flow in that vertical is sampled proportionately to the velocity.

The open-mouth weighted-bottle sampler, therefore, does not collect a truly representative sample in a flowing stream if there are many particles coarser than about 0.062 mm carried in suspension (*17*). Another disadvantage to using an open-mouth bottle sampler in flowing streams is that there is no assurance when the bottle was filled, compounding the uncertainty that the sample collected truly represented the distribution of both dissolved and suspended material in the sampled water column. This method of sampling may be extremely poor for flowing streams but used effectively for slow-moving bodies of water, lakes, and estuaries.

Recent research studies of flow and sediment transport in the Rio Grande conveyance channel near Bernardo, N. Mex., a typical sand-bed channel (*4*), illustrates variability in the vertical and lateral distribution of sediment in a cross section. Velocity was measured (*2, 3*), and suspended-sediment samples were collected (*8,18*) at five points in each of six verticals in a cross section. Sediment samples were collected using a standard depth-integrating, suspended-sediment sampler modified to collect point integrated samples. Particle-size distributions were determined using standard laboratory, fall-velocity techniques. Concentration of sediment in the following size classes was determined for each sample: silt and clay (finer than 0.062 mm) and sand (between 0.062 and 2.0 mm).

Fig. 2 shows the velocity distribution in the 80-ft wide cross section. There were four cells of high velocity, resulting largely from the turbulent characteristics of the dune-bed form. The highest velocity was near mid-channel and the lowest velocity was along the right bank.

The distribution of concentration of the silts and clays is shown in Fig. 3. The open circles indicate the location of the six sampled verticals. Concentration values (mg/liter) are shown at the top of the figure. The upper values are the concentrations at the surface, and the lower values are the mean concentration in the vertical. The differences are evident; the surface samples generally were lower than the mean in the verticals, and in this case, averaged about 4% less than the mean in the cross section. The concentration at the surface of the right bank was the lowest in the cross section, coinciding with the low-velocity zone, and was about 92% of the mean in the cross section. A dip sample taken from the right bank would reflect an 8% minimum error with respect to fine suspended material.

Distribution of the concentration of sand is shown in Fig. 4. The distribution of sand is quite different from the distribution of silt and clay. Concentrations at the surface generally were about one-half the mean concentration in the verticals, and the concentration at the surface at the right bank was only about one-half the concentration at the surface at the left bank. The solid circles represent depths at which samples have to be collected to sample the mean concentration of sand in that vertical. In this case, which is rather typical in sand-channel streams, a surface sample collected along the right bank would represent only 26% of the mean in the cross section and is not representative of all sizes of particles in transport. A sample collected along the left bank would be about 47% of the mean. Thus, a real sampling problem related to sand in transport is apparent.

One sampling technique currently accepted by hydrologists for use in such situations is the equal-transit-rate method (ETR) (*8*). In this method, standard suspended-sediment samplers are used to collect a velocity-weighted

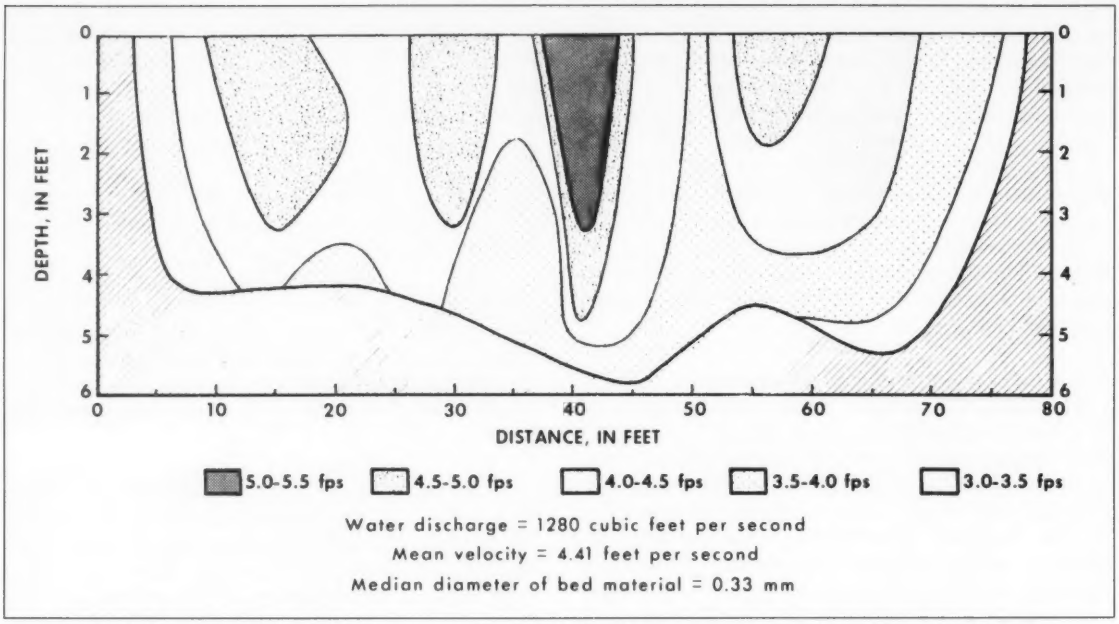

FIGURE 2.—*Velocity distribution in cross section of Rio Grande conveyance channel near Bernardo, N. Mex.*

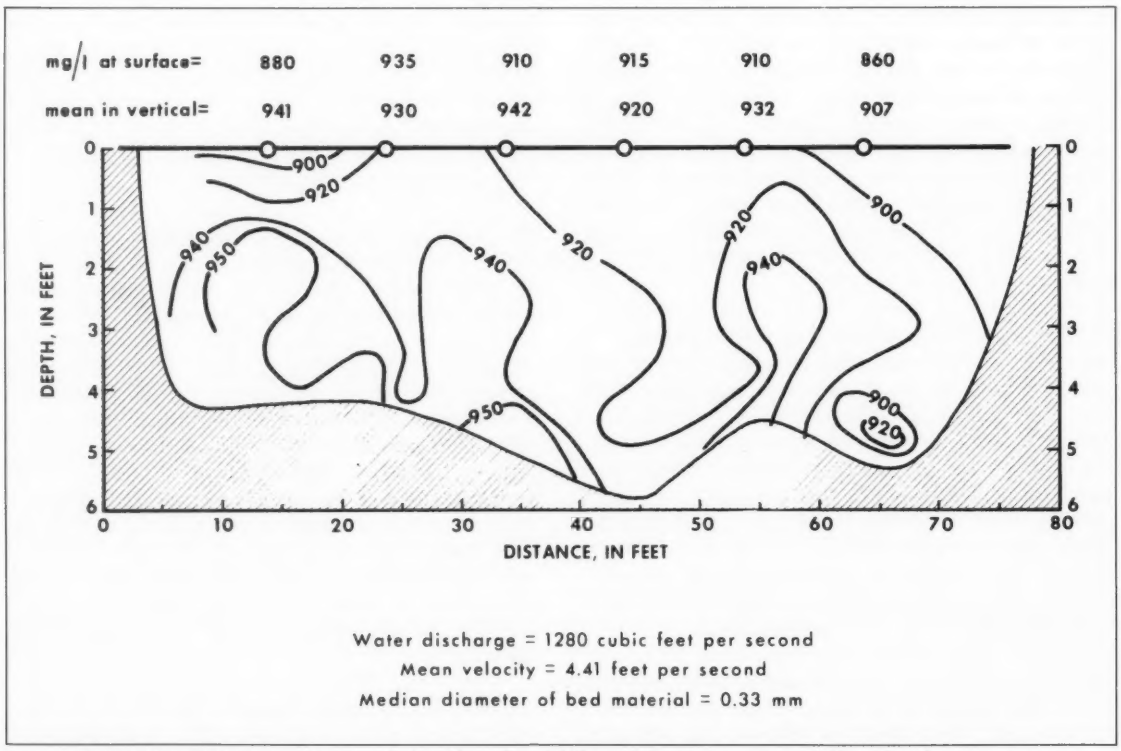
mg/l at surface= 880 935 910 915 910 860
mean in vertical= 941 930 942 920 932 907
DEPTH, IN FEET
DISTANCE, IN FEET
Water discharge = 1280 cubic feet per second
Mean velocity = 4.41 feet per second
Median diameter of bed material = 0.33 mm

FIGURE 3.—*Distribution of silt and clay (finer than 0.062 mm) in cross section of Rio Grande conveyance channel near Bernardo, N. Mex.*

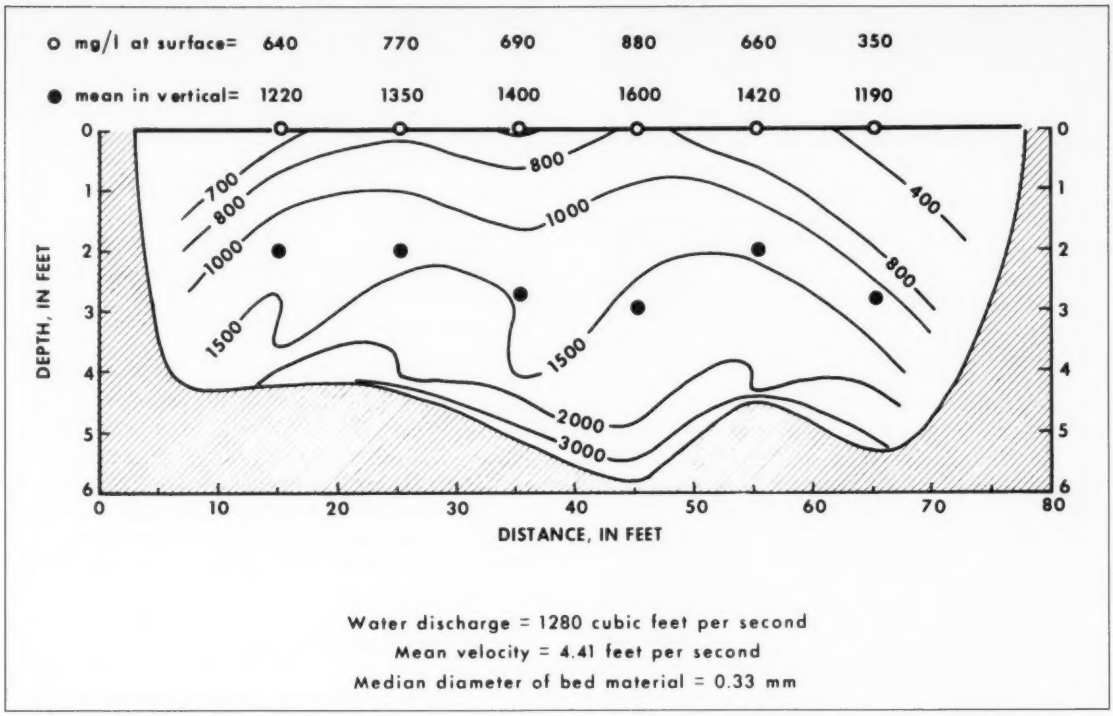
o mg/l at surface= 640 770 690 880 660 350
● mean in vertical= 1220 1350 1400 1600 1420 1190
DEPTH, IN FEET
DISTANCE, IN FEET
700
800
1000
1500
2000
3000
400
Water discharge = 1280 cubic feet per second
Mean velocity = 4.41 feet per second
Median diameter of bed material = 0.33 mm

FIGURE 4.—*Distribution of sand (between 0.062 mm and 2.0 mm) in cross section of Rio Grande conveyance channel near Bernardo, N. Mex.*

sample. Samples are taken at a number of equally spaced verticals in the cross section depending on stream width. The transit rate of the sampler, which is the rate of movement of the sampler from the water surface to the stream bed and back to the surface, is the same at all verticals. Samples collected in each vertical are all composited into a single sample representative of the entire flow in the cross section. In this manner, the composite sample of the water-sediment mixture flowing in the cross section is velocity- and discharge-weighted.

Traditional dip samples, collected at the six points in the cross section and composited, would have contained concentrations of suspended sediment representative of about 96% of the silts and clays but only about 49% of the sands being transported. The discharge of silts and clays was at the rate of 3,200 tons per day, and sand at 4,700 tons per day; thus, the dip samples would have been representative of only 3,070 and 2,300 tons per day for fine and coarse material, respectively. Pesticides associated with about 32% of the suspended sediment in transport would not have been accounted for.

The significance of the sediment-distribution data presented is that without prior knowledge of the hydraulic and sediment transport conditions at a given site, gross sampling errors may accrue. There is no justification in striving for 5% allowable error in the laboratory analysis of poorly collected samples that may represent only ±30-40% of the true water-sediment mixture. It is incumbent upon all investigators to recognize that pesticide concentrations currently reported for streams can reflect gross errors, and that these values may be extremely misleading in any assessment of residue distribution. Although a western sand-bed stream has been used for illustration, the authors recommend that the ETR sampling technique described herein should be used in all flowing streams.

BOTTOM MATERIALS

Pesticides associated with bottom material, irrespective of the ratio of inorganic to organic composition, may reflect an integration of chemical and biological processes. They serve the indispensable historical role by reflecting input to non-scouring streams, lakes, and esturaries with respect to time, application of pesticides, and land use. Recalling that fluvial materials tend to settle out during periods of low streamflow or of calm conditions in lakes and are additive to solids that have accumulated on stream beds or in lake bottoms, periodic

sampling of water overlying these deposits might not reveal the presence of pesticides, even though they may be present in the solid material which acts as a sink and reservoir.

The loss of low-density deposits must be kept minimal during any sampling process, requiring a bottom-material sampler that is capable of collecting and retaining the "fines" which sometimes contain the highest concentration of pesticide residues.

Few data have appeared in the literature regarding the presence of pesticide residues at the liquid-solid interface or in the hydrosol area. Depending upon ambient physical, biological, and chemical controls, residues may be transformed into metabolites, degraded, or taken into solution. Further study of these processes is an open and challenging field of endeavor. An evaluation of this portion of the hydrosystem is essential to comprehensive assessments of the occurrence and distribution of pesticides.

Sample Collection and Preservation

To overcome the problems associated with collecting representative samples, equipment that has been specifically designed and thoroughly tested is favored. There are several depth-integrating samplers available that are suitable. In shallow streams and wetlands that can be waded, the US DH-59 suspended-sediment sampler (Fig. 5) can be used successfully. The sampler is simple and of clean design. A sample container is easily inserted into position and held firmly by spring action.

The US D-49 suspended-sediment sampler (Fig. 6) has been used for many years in collecting depth-integrated suspended-sediment samples in the larger streams and rivers. It is provided with a choice of nozzles (shown beneath the tail fins), ⅛-, 3/16-, and ¼-inch in diameter, with which inflow of the water-sediment mixture can be controlled. The sampler, which weighs about 60 pounds, is suspended on a cable and operated with a reel attached to a boom. The open-mouth, weighted-bottle sampling technique used in the National Pesticide Monitoring Program is acceptable for sluggish streams, lakes, and reservoirs (*5, 7*).

Bottom deposits present a more difficult set of sampling conditions than those in flowing streams or in lakes, primarily because of the varying firmness of bed materials. The US BMH-60 bed-material sampler (Fig. 7). is a 30-pound impact type sampler designed primarily for use in sand-bed streams. However, it works equally well for firm and partly consolidated bottom materials. One man can collect samples with this sampler under most conditions. The bucket takes a bed-material sample that is approximately 2.2 x 5 x 1.7 inches. This sampler can be used in about 75% of the river miles across the United States. It also can be used in many lakes and reservoirs to collect excellent samples (Fig. 8). The equipment is described in detail in references *8* and *18*.

Numerous investigators prefer to collect core samples when interest is primarily in historical data and when the bed is sufficiently compacted to be sampled with a coring device. The value of this type of sample should not be minimized; however, fresh deposition is of prime importance in a continuous evaluation of the occurrence, distribution, and movement of pesticide residues.

Most core samplers lack positive seals to hold a core or moist sample in place as the sampler is withdrawn from the water. The Controlled-Depth Volumetric Bottom Sampler described by Jackson (*9*), and the Core Sampler for *in situ* Freezing of Benthic Sediments designed by Gleason and Ohlmacher (*6*) are examples of efforts to overcome the drawbacks of the common core sampler and to provide the investigator with reliable equipment.

Little progress has been made in developing equipment to collect representative samples at the liquid-solid interface because it is difficult to sample without disturbance. The Gleason core sampler offers an advantage in sampling this interface because it can be used as a point sampler. The Hydrosol Sampler designed by Lawrence (*14*) may be used in many sampling situations. Reporting of experiences in using these and other hydrosol samplers will help to determine the adequacy of the samplers.

Deteriorated samples negate all the effort and cost expended in obtaining good samples. Samples of water plus suspended material should be forwarded to the laboratory in cleansed glass containers by the fastest transportation available. Bottom materials can be placed in the same type of container as suspended-sediment samples when they have a high percentage of water; solid bed-material samples may be sealed in foil. All samples should be kept near freezing, and extractions should be carried out in minimum elapsed time, preferably within 2 days from time of sample collection. Fresh plant material should be wrapped in foil and kept chilled or frozen; fauna should be wrapped in foil and frozen.

Sampling Frequency

In conducting reconnaissance studies, defined here as short-term, one-time evaluations, both bottom deposits and the overlying water should be sampled at each site. Monitoring, which consists of repetitive, continuing measurements to define variations and trends at a given

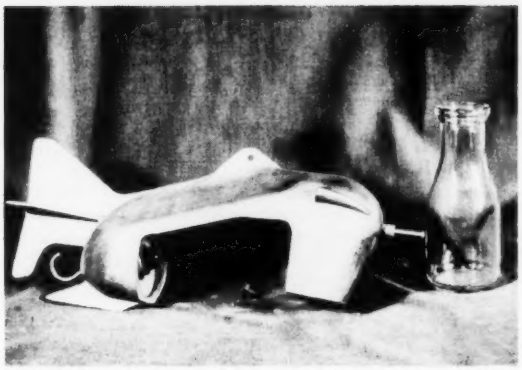

FIGURE 5.—*US DH-59 suspended-sediment sampler*

FIGURE 6.—*US D-49 suspended-sediment sampler*

location, should include collection of water samples during each of the four seasons, with particular emphasis on the fall and spring collections. Most flood events should be sampled. Bed-material samples should be collected for analysis of fresh deposits at least once per year at monitoring sites, and preferably during both the spring and fall seasons (*5*).

Evaluation of the variability in available pesticide data must precede any decision as to the number of samples and collection frequency required to maintain an effective monitoring program.

Analytical Schedule

To use fully all reported data, analytical and quality-control procedures must be agreed upon and standardized. Data contributions by all investigators constitute what may be regarded as a National Data Bank. Each piece of information should be virtually equivalent in terms of accuracy and reliability. To insure this condition, analytical schedules should be derived from consideration of the persistence, toxicity, and total usage of pesticides, the number of reported positive identifications, and the listings in the Revised Chemicals Monitoring Guide for the National Pesticide Monitoring Program (*16*). Schedules must be flexible and responsive to local problems. All significant peaks on chromatograms should be identified and quantified, if possible, and a report made of products that are pesticidal or toxic. Identification and quantification by dual-column electron-capture gas chromatography should be considered minimal. Back-up tools are essential, and confirmations can be made by use of a third column with diverse retention times, thin layer chromatography, column chromatography, microcoulometry, flame-photometric detection, infrared, and mass spectrometry (*5*).

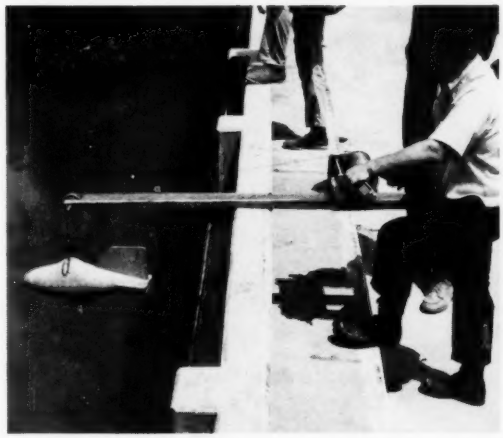

FIGURE 7.—*US BMH-60 bed-material sampler in use on South River below Atlanta, Ga.*

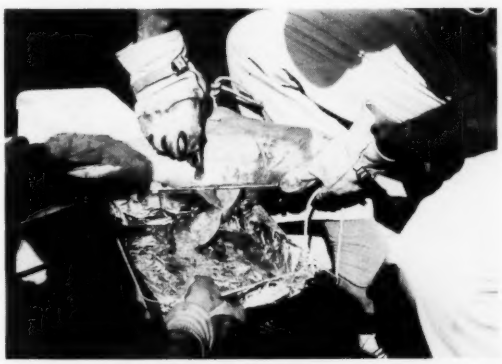

FIGURE 8.—*Bottom material being released from BMH-60 sampler*

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Organochlorine Insecticides in Surface Waters in Germany—1970 and 1971 [1]

Fritz Herzel

ABSTRACT

As part of a series of studies initiated in 1969 to determine the organochlorine insecticide content of major waters in the Federal Republic of Germany, unfiltered water and suspended solids were analyzed from approximately 25 sites sampled in May 1971, and unfiltered water was analyzed from 7 sites sampled monthly from April 1970—June 1971. As in former studies (June and October 1969, April and September 1970), the insecticide concentrations found in waters and suspended solids were almost exclusively in the ppt range (ng/liter).

The compounds found most frequently were γ-BHC (lindane) and α-BHC; α- and β-endosulfan were detected in the Main, Regnitz, and Rhine Rivers. DDT and particularly its metabolites DDD and DDE were found infrequently except in samples from the Berlin Teltowkanal. Findings of heptachlor, heptachlor epoxide, dieldrin, and parathion (the only organophosphorus insecticide included in the study) were rare.

Introduction

Extensive surveys have been carried out to determine the presence of chlorinated pesticides in surface waters (*1-14, 18-36*); however, until recent years studies of this type had not been conducted in the Federal Republic of Germany. To obtain data on the organochlorine insecticide content of surface waters in the Federal Republic, a series of studies was initiated in 1969. The overall program consisted of the analysis of water and suspended solids or sediment samples obtained during five sampling excursions and water samples collected each month over the period April 1970-June 1971. The five sampling excursions which extended, as a rule, over a period of 1 week involved sampling the major bodies of water in the Federal Republic at approximately 25 preselected sites. Because some organochlorine insecticides are almost insoluble in water but will adsorb to suspended matter, in addition to the analysis of water samples, suspended solids were filtered from other water samples collected at most of these same sites and analyzed separately. The sampling excursions were as follows:

(1) June 1969—water only, with additional sampling at some sites which had been designated as potentially endangered, e.g., waters in fruit- and hop-growing areas, agricultural drainage ditches, etc.;

(2) October 1969—water and suspended solids;

(3) April 1970—water and sediment;

(4) September 1970—water and suspended solids; and

(5) May 1971—water and suspended solids.

Because of unfavorable weather conditions, a sixth sampling excursion scheduled for the winter months did not take place.

Results from the first four sampling excursions have been published elsewhere (*15, 16*). This paper presents the results of the May 1971 excursion during which approximately 25 sites were sampled for water and suspended sediments; data are also reported on the analysis of water samples collected monthly from 7 sites during the period April 1970-June 1971. Specific locations of sampling sites are shown on Fig. 1.

Sampling Procedures

One of the most difficult problems in sampling surface waters is obtaining a representative sample, particularly in flowing waters, since concentrations vary considerably

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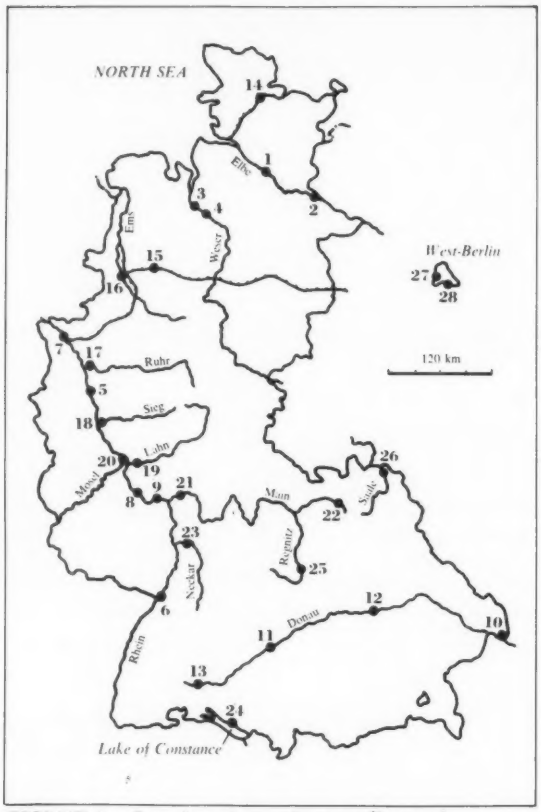

FIGURE 1.—*Location of sites for sampling surface waters in the Federal Republic of Germany*

with time in a downstream direction as well as horizontally and vertically across the stream. Theoretically, these variations can be overcome with an ideal device for continuous sampling; however, the general experience of this investigator with continuous sampling devices has been unfavorable, because they did not collect suspended matter in a uniform proportion to water collected.

WATER SAMPLES

Water samples were collected in 1-liter round-bottom flasks attached with a clamp to the end of a 2-m steel rod. The flasks were filled by moving them evenly up and down at an approximate depth of 50 cm. Before this, 0.5 ml toluene and 30 mg mercuric chloride had been added to the flasks as preservatives for the water samples; the flasks were stored in heat-insulation fitted transport boxes of foamed polystyrene, and sent to the laboratory with as little delay as possible.

SUSPENDED SOLIDS

Water from which suspended solids were obtained was collected in a steel bucket that was cleaned between samplings with alcohol. Suspended solids were filtered from the water using a Buchner funnel (32 cm in diameter) fitted with filter paper (Selecta No. 1450) that had been carefully pre-extracted with acetone. The water was drawn through the filter into a 10-liter suction flask and a vacuum pump operated by a portable small-size generator until the filter was filled to capacity. The paper filters holding the filtration residues were folded and put into 100-ml flasks each containing 20 ml of a 1:1 mixture of hexane and acetone to reduce biological degradation.

An effort was made to select sampling sites with sufficient depth to avoid stirring of sediment at the bottom, particularly where samples were taken with a bucket. In several cases, however, the water was too shallow and samples for analysis of suspended solids could not be collected. Sampling of water for water analysis (using flasks) and water for obtaining suspended solids (using buckets) took place at the same site whenever possible; however, bank conditions did not permit this in all instances.

Analytical Procedures

PREPARATION OF SAMPLES

Water Samples

Water samples were extracted in a separatory funnel with subsequent quantities of 15, 10, and 10 ml of hexane, (b.p. 68-70° C, column distilled). The extracts were then combined and without cleanup (*17*) were analyzed by electron capture gas-liquid chromatography (EC-GC).

Suspended Solids (Filtration Residues)

Solvents used in extraction and cleanup of suspended solids were hexane, b.p. 68-70° C; acetone, reagent grade; and benzene, reagent grade—each column distilled.

Suspended solids, which had been collected on filters and placed in a mixture of acetone and hexane, were extracted with an additional 50 ml of a 1:1 mixture of acetone-hexane in a Soxhlet apparatus, and poured into 5 times their volume of water. The nonpolar phase was separated, reduced, and then cleaned up on a Florisil column, 20 cm by 10 mm, i.d., filled with 7 cm of Florisil, 60/100 mesh, and 1 cm of anhydrous sodium sulphate on top; the Florisil had been prepared by heating at 130° for 16 hours and 3% w/w of water added, and the anhydrous sodium sulphate by heating at 400° C for 16 hours. The column was prewetted with hexane, and the samples were eluted with 30 ml of a 1:1 mixture of hexane and benzene. First, these eluates were analyzed by EC-GC. Then, they were evaporated to dryness in a rotary vacuum evaporator

and taken up in 0.1 ml amylacetate for subsequent microcoulometric gas chromatography and thin layer chromatography. The extracts from the water samples were treated likewise after their EC-GC detection.

Previous experience has shown that in using a rotary vacuum evaporator, there is no risk of volatilization of insecticide from extracts even if complete reduction to dryness takes place. Even in pure solutions, as contrasted with air drying, there were no notable losses when the procedure took place at room temperature and when the plunger was removed from the vacuum as soon as the solvent had evaporated. To insure that there was no loss of insecticides, a few milliliters of decyl acetate was added.

GAS CHROMATOGRAPHY

Gas chromatographic determination was carried out on Aerograph gas chromatographs, models 205-B and 1740, equipped with two electron capture detectors (tritium) and, additionally, a microcoulometric detector for confirmation. The operating parameters were as follows:

Columns:	Glass, ⅛" by 5' packed with 2-3% Dow-11, OV-101, XE-60, and Dexsil 300 on 80/100 mesh Chromosorb W, AW, DMCS treated
Temperatures:	Injector—210° C Column—185° C Detector—195° C
Recorder:	1 mv full scale deflection

The detection limits of organochlorine insecticides in 30 ml of extract were between 5 and 10 ng for hexachlorobenzene (HCB) and the hexachlorocyclohexane isomers α- and γ-BHC (lindane), 100-200 ng for DDT and its derivatives, and 10-30 ng for the remaining organochlorines. In a number of cases, particularly DDT, the sample was concentrated to one-tenth of the original volume, thus lowering the detection limit by one power of ten.

The detection limit for parathion was 1 ng; quantitative determination was based on the quantity of 0.1-ml amylacetate which had been used, as described above, to take up the residue from the 30-ml hexane extract. An alkali FID was employed for quantitative gas chromatographic determination of parathion because of its comparatively low susceptibility in respect to high concentrations of interfering substances in a concentrated extract of this type. Since the concentration per liter of parathion in suspended solids was based on the amount of residue detected divided by the number of liters of water filtered, very low concentrations could be detected.

RECOVERY

The recovery rates from water samples were checked repeatedly: reproducibility was quite good, at rates exceeding 90% in all cases. The recovery values of residues in suspended solids varied between 80% and 95%, depending on the insecticide residue. Results were not corrected for percent recovery.

CONFIRMATION STUDIES

In doubtful cases, preseparation by thin layer chromatography (TLC) was used for further confirmation of results. After the volume of the extract had been reduced to dryness and then taken up by 0.1 ml of amyl acetate, approximately one-half the amount (0.05 ml) was placed on 20- by 20-cm TLC plates coated with 0.3-mm silica gel (Kieselgel HF Merck) which had been dried 1 hour at 105° C. Hexane was used as the solvent, and the suspected substances served as references. After removal of the corresponding zone and elution by 1 ml of benzene, gas chromatographic analysis using an electron capture detector was repeated.

As a rule, a particular insecticide was considered present only if corresponding peaks were obtained from all GC columns; therefore, a major number of peaks were considered unidentifiable. Preseparation by TLC was performed in addition when the comparatively low microcoulometric reading of less sensitive substances like DDT was obtained. The polychlorinated biphenyls were not considered present, however, without confirmation by mass spectrometry even where typical fingerprints were observed. These compounds have not been detected in any samples taken during this series of studies.

Results and Discussion

Results of analyses of unfiltered water and suspended solids are given in Tables 1 and 2. Residue levels in water are expressed as ng/liter (ppt) and values obtained from suspended solids as nanograms of insecticides in solids suspended in 1 liter of water, i.e., the quantities obtained from the filter residues were divided by the number of liters of water that had been filtered.

Generally, insecticide concentrations were low in both types of samples. In water samples, heptachlor, heptachlor epoxide, and dieldrin were found seven times, three times, and once, respectively. In the suspended solids heptachlor was detected only once, and aldrin, dieldrin, and heptachlor epoxide were not found in any of the samples. The results were similar to those from earlier studies. Concentrations of endosulfan, however, were clearly lower as compared with earlier studies; residues of this compound, originating from industrial effluents, were found in samples from the Rhine, the lower Main (downstream from its junction with the Rhine), and the Regnitz.

The insecticides found most frequently were the hexachlorocyclohexane isomers, α-BHC and lindane (γ-BHC); however, the proportion of these compounds

found in suspended solids was low because of their greater solubility in water. Because of technical difficulties, only a few samples were analyzed for α-BHC and hexachlorobenzene (HCB), and concentrations were determined with certainty only in some samples of suspended solids. The results obtained, however, showed a notable increase of lindane and α-BHC concentrations in samples from the upper Rhine and at the Berlin Teltowkanal over values obtained in previous studies.

DDT and its main metabolites, DDD and DDE, occupied a subordinate rank with regard to the frequency of positive findings and concentrations in waters. Because of extremely low solubility in water, DDT was found mainly adsorbed to suspended solids.

Parathion, the only organophosphate analyzed in this study, was found in only a few samples and in very small amounts.

In evaluating the degree of pollution of major German surface waters by organochlorine insecticides, results obtained in this study as well as former studies show that the Rhine River is the most contaminated.

Acknowledgment

A great number of samples were furnished regularly from various sources. The author wishes to express his gratitude, in particular, to the Hygienisches Institut, Hamburg; Wasserwerke Bremen AG; the Düsseldorf Field Station of this Institute; the Institut für Gastechnik, Feuerungstechnik und Wasserchemie, Technical University Karlsruhe, and Regierung von Niederbayern, Abt. Gewässergüteaufsicht, Landshut. Special thanks for technical assistance are expressed to Mrs. H. Junge and Mrs. F. Reichert.

See Appendix for chemical names of compounds discussed in this paper.

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TABLE 1.—*Concentrations of insecticides in unfiltered surface waters in the Federal Republic of Germany—May 1971 sampling excursion and monthly collections from April 1970-June 1971*

[blank = not analyzed; — = not detected]

Sampling Site No. (See Fig. 1)	Body of Water	Location	Date of Sampling	Insecticide Concentrations in ng/liter (ppt)										
				α-BHC	Lindane	Heptachlor	Heptachlor epoxide	Dieldrin	α-Endosulfan	β-Endosulfan	DDE	DDD	DDT	Parathion
1	Elbe	Hamburg	5/21/70	—	215	—	—	—	—	—	—	—	—	—
			6/11/70		250	—	—	—	—	—	—	—	—	—
			7/23/70		395	—	—	—	—	—	—	—	—	—
			8/70		310	—	—	—	—	—	—	—	—	—
			9/01/70		175	—	—	—	—	—	—	—	—	—
			10/28/70		125	—	—	—	—	—	—	—	—	—
			11/05/70		160	—	—	—	—	—	—	—	—	—
			12/14/70		185	20	—	—	—	—	—	—	—	—
			1/27/71	810	255	—	—	—	—	—	—	—	—	—
			2/25/71	1500	430	—	—	—	—	—	—	—	—	—
			5/03/71	1050	275	—	—	—	—	—	—	—	—	—
			6/15/71	690	250	—	—	—	—	—	—	—	—	—
2	Elbe	Lauenburg	5/10/71	—	145	—	25	—	—	—	—	—	165	—
3	Weser	Bremen	4/13/70	—	10	—	—	—	—	—	—	—	—	—
			5/11/70	—	5	—	—	—	—	—	—	—	—	—
			6/08/70		30	—	—	—	—	—	—	—	—	—
			7/13/70		25	—	—	—	—	—	—	—	—	—
			8/10/70		30	—	—	—	—	—	—	—	—	—
			9/07/70		20	—	—	—	—	—	—	—	—	—
			10/12/70		25	10	—	—	—	—	—	—	—	—
			11/09/70		30	—	—	45	—	—	—	—	—	—
			12/07/70		5	—	—	—	—	—	—	—	—	—
			1/11/71	—	5	—	—	—	—	—	—	—	—	—
			2/08/71	—	10	—	—	—	—	—	—	—	—	—
			3/08/71	—	5	—	—	—	—	—	—	—	—	—

TABLE 1.—*Concentrations of insecticides in unfiltered surface waters in the Federal Republic of Germany—May 1971 sampling excursion and monthly collections from April 1970-June 1971*—Continued

Sampling Site No. (See Fig. 1)	Body of Water	Location	Date of Sampling	Insecticide Concentrations in ng/liter (ppt)										
				α-BHC	Lindane	Heptachlor	Heptachlor epoxide	Dieldrin	α-Endosulfan	β-Endosulfan	DDE	DDD	DDT	Parathion
3	Weser	Bremen	4/13/71	—	30	—	—	—	—	—	—	—	—	—
			5/10/71	—	50	—	—	—	—	—	—	—	—	—
			6/14/71	—	60	—	—	—	—	—	—	—	—	—
4	Weser	Achim	5/10/71	25	45	—	—	—	—	—	—	—	—	—
5	Rhine	Düsseldorf	4/70	720	245	—	—	—	70	—	—	—	—	—
			6/70		215	—	—	—	—	—	—	—	—	—
			7/70		105	—	—	—	—	—	—	—	—	—
			8/70		165	—	—	—	55	95	—	—	—	—
			9/70		105	—	—	—	35	—	—	—	—	—
			10/70		140	—	—	—	15	20	—	—	170	—
			11/70		100	—	—	—	15	—	—	—	—	—
			12/70		220	—	—	—	—	—	—	—	—	—
			1/71	905	115	205	—	—	—	—	—	—	—	55
			2/71	555	175	—	—	—	—	—	—	—	—	5
			3/71	125	110	—	—	—	—	—	—	—	—	—
6	Rhine	Karlsruhe	4/15/70	1900	380	—	—	—	—	—	—	—	—	—
			5/14/70	155	130	—	—	—	—	—	—	—	—	—
			6/70		50	—	—	—	—	—	—	—	—	—
			7/15/70		60	—	—	—	—	—	—	—	—	—
			8/10/70		225	—	—	—	—	—	—	—	—	—
			9/70		455	—	—	—	—	—	—	—	—	—
			10/13/70		125	—	—	—	—	—	—	—	55	—
			11/09/70		265	—	—	—	—	—	—	—	—	—
			12/10/70		175	—	—	—	—	—	—	—	—	—
			1/13/71	1000	325	—	—	—	—	—	10	—	—	—
			2/71	945	330	—	—	—	—	—	—	—	—	5
			3/16/71	1100	330	—	—	—	—	—	—	—	—	—
			4/71	1300	260	—	—	—	—	—	—	—	—	—
			5/71	2400	535	—	—	—	—	—	—	—	—	—
			5/12/71	890	240	—	—	—	—	—	—	—	—	—
7	Rhine	Wesel	5/11/71	115	155	—	—	—	—	—	—	—	—	—
8	Rhine	St. Goar	5/11/71	110	260	—	—	—	—	—	—	—	—	—
9	Rhine	Oestrich	5/12/71	550	130	—	—	—	55	—	—	—	—	—
10	Danube	Jochenstein	4/15/70	—	5	—	—	—	—	—	—	—	—	—
			5/13/70	5	20	—	—	—	—	—	—	—	—	—
			6/10/70		30	—	—	—	—	—	—	—	—	—
			7/08/70		25	—	—	—	—	—	—	—	—	—
			8/19/70		20	—	—	—	—	—	—	—	—	—
			9/16/70		25	—	—	—	—	—	—	—	—	—
			10/14/70		15	—	—	—	—	—	—	—	—	—

TABLE 1.—*Concentrations of insecticides in unfiltered surface waters in the Federal Republic of Germany—May 1971 sampling excursion and monthly collections from April 1970-June 1971*—Continued

Sampling Site No. (See Fig. 1)	Body of Water	Location	Date of Sampling	Insecticide Concentrations in ng/liter (ppt)										
				α-BHC	Lindane	Heptachlor	Heptachlor epoxide	Dieldrin	α-Endosulfan	β-Endosulfan	DDE	DDD	DDT	Parathion
10	Danube	Jochenstein	11/11/70		45	—	—	—	—	—	—	—	—	—
			12/09/70		35	—	—	—	—	—	—	—	—	—
			1/20/71	—	5	—	—	—	—	—	—	—	—	—
			2/17/71	—	30	—	—	—	—	—	—	—	—	—
			3/17/71	—	15	—	—	—	—	—	—	—	—	—
			4/14/71	—	30	—	—	—	—	—	—	—	—	—
			5/12/71	—	5	—	—	—	—	—	—	—	—	—
			6/09/71	50	40	—	—	—	—	—	—	—	—	—
11	Danube	Ulm	5/13/71	—	25	—	—	—	—	—	—	—	—	—
12	Danube	Ingolstadt	5/14/71	40	30	—	—	—	—	—	—	—	—	—
13	Danube	Geisingen	5/13/71	20	40	—	—	—	—	—	—	—	—	—
14	Nord-Ostsee-Kanal	Rendsburg	5/10/71	—	—	—	—	—	—	—	—	—	—	—
15	Mittelland-Kanal	Bramsche	5/11/71	—	15	—	35	—	—	—	—	—	—	—
16	Ems	Rheine	5/11/71	—	—	—	—	—	—	—	—	—	—	—
17	Ruhr	Duisburg	5/11/71	—	—	—	—	—	—	—	—	—	—	—
18	Sieg	Siegburg	5/11/71	295	615	—	—	—	—	—	—	—	—	—
19	Lahn	Fachbach	5/11/71	—	—	—	—	—	—	—	—	—	—	—
20	Moselle	Koblenz	5/11/71	25	20	—	—	—	—	—	—	—	—	—
21	Main	Raunheim	5/12/71	170	64	—	—	—	100	45	—	—	—	—
22	Main	Bad Berneck	5/14/71	5	30	—	—	—	—	—	—	—	—	—
23	Neckar	Heidelberg	5/12/71	—	—	—	—	—	—	—	—	—	—	—
24	Lake Constance	Langenargen	5/13/71	40	25	—	—	—	—	—	—	—	—	—
25	Regnitz	Erlangen	5/14/71	170	140	20	40	—	10	—	—	—	100	—
26	Saale	Hof	5/15/71	95	55	—	—	—	—	—	—	—	—	—
27	Havel	Berlin-Gatow	4/29/70	10	10	—	—	—	—	—	—	—	—	—
			5/22/70	—	5	—	—	—	—	—	—	—	—	—
			6/24/70		50	—	—	—	—	—	—	—	—	—
			7/08/70		80	—	—	—	—	—	—	—	—	—
			8/12/70		40	—	—	—	—	—	—	—	—	—
			9/17/70		55	—	—	—	—	—	—	—	—	—
			10/27/70		20	—	—	—	—	—	—	—	45	—
			11/02/70		45	—	—	—	—	—	—	—	—	—
			12/01/70		80	15	—	—	—	—	—	—	—	—
			1/21/71	5	70	—	—	—	—	—	—	—	—	—
			2/10/71	5	5	—	—	—	—	—	—	—	—	—
			3/25/71	95	25	25	—	—	—	—	—	—	—	—
			4/21/71	165	50	30	—	—	—	—	—	—	—	—
			5/12/71	190	70	—	—	—	—	—	—	—	—	—
			6/21/71	115	55	—	—	—	—	—	—	—	—	—

TABLE 1.—*Concentrations of insecticides in unfiltered surface waters in the Federal Republic of Germany—May 1971 sampling excursion and monthly collections from April 1970-June 1971*—Continued

Sampling Site No. (See Fig. 1)	Body of Water	Location	Date of Sampling	Insecticide Concentrations in ng/liter (ppt)										
				α-BHC	Lindane	Heptachlor	Heptachlor epoxide	Dieldrin	α-Endosulfan	β-Endosulfan	DDE	DDD	DDT	Parathion
28	Teltow-Kanal	Berlin-Lichterfelde	4/29/70	135	170	—	—	—	—	—	—	—	—	—
			5/11/70	515	950	—	—	—	—	—	—	—	—	—
			6/24/70		370	—	—	—	—	—	—	—	65	—
			7/08/70		420	—	—	—	—	—	—	250	—	—
			8/12/70		190	—	—	—	—	—	—	95	—	—
			9/17/70		450	—	—	—	—	—	—	190	—	—
			10/27/70		60	—	—	—	—	—	—	80	60	—
			11/02/70		1350	—	—	—	—	—	35	180	—	—
			12/01/70		7100	—	—	—	—	—	85	—	135	—
			1/21/71	415	1650	—	—	—	—	—	—	85	—	—
			2/10/71	1700	800	—	—	—	—	—	—	130	—	65
			3/25/71	400	1750	—	—	—	—	—	—	200	—	—
			4/21/71	530	715	—	—	—	—	—	—	830	—	—
			5/12/71	545	540	—	—	—	—	—	70	775	—	—
			6/21/71	1245	80	—	—	—	—	—	—	—	—	—

TABLE 2.—*Concentrations of insecticides in suspended solids from surface waters in the Federal Republic of Germany—May 1971*

[— = not detected]

Sampling Site No. (See Fig. 1)	Body of Water	Location	Date of Sampling	Liters of Water Filtered	Insecticide Concentrations in ng/Solids Suspended in 1 Liter of Water (ppt)[1]							
					HCB	α-BHC	Lindane	Heptachlor	α-Endosulfan	β-Endosulfan	DDT [2]	Parathion
2	Elbe	Lauenburg	5/10/71	18	57	—	37	—	—	—	36	—
4	Weser	Achim	5/10/71	14	—	—	53	—	—	—	—	—
6	Rhine	Karlsruhe	5/12/71	18	—	2.5	21	—	—	—	—	—
7	Rhine	Wesel	5/11/71	12	—	—	53	—	—	—	—	0.4
8	Rhine	St. Goar	5/11/71	21	—	37	22	—	—	—	—	—
9	Rhine	Oestrich	5/12/71	12	36	42	39	—	—	—	—	0.15
11	Danube	Ulm	5/13/71	34	—	—	0.4	—	—	—	—	—
12	Danube	Ingolstadt	5/14/71	24	0.6	—	—	2	—	—	4.4	—
14	Nord-Ostsee-Kanal	Rendsburg	5/10/71	32	—	—	—	—	—	—	—	—
15	Mittelland-Kanal	Bransche	5/11/71	13	—	—	3.1	—	—	—	—	—
16	Ems	Rheine	5/11/71	12	—	—	0.8	—	—	—	—	—

TABLE 2.—*Concentrations of insecticides in suspended solids from surface waters in the Federal Republic of Germany—May 1971*—Continued

Sampling Site No. (See Fig. 1)	Body of Water	Location	Date of Sampling	Liters of Water Filtered	Insecticide Concentrations in ng/Solids Suspended in 1 Liter of Water (ppt)[1]							
					HCB	α-BHC	Lindane	Heptachlor	α-Endosulfan	β-Endosulfan	DDT[2]	Parathion
17	Ruhr	Duisburg	5/11/71	20	—	—	12	—	—	—	—	—
18	Sieg	Siegburg	5/11/71	20	—	—	—	—	—	—	—	—
19	Lahn	Fachbach	5/11/71	12	1	—	2.1	—	—	—	8.7	—
20	Moselle	Koblenz	5/11/71	25	0.6	—	1.2	—	—	—	4.2	—
21	Main	Raunheim	5/12/71	13	19	25	18	—	22	9.6	264	0.15
23	Neckar	Heidelberg	5/12/71	32	—	—	0.15	—	—	—	—	—
24	Lake Constance	Langenargen	5/13/71	25	—	—	0.4	—	—	—	—	—
25	Regnitz	Erlangen	5/14/71	11	21	—	159	—	24	—	119	—
26	Saale	Hof	5/15/71	13	6.5	—	6.9	—	—	—	31	—

NOTE: Aldrin, dieldrin, and heptachlor epoxide were not detected in samples of suspended matter.
[1] Quantities obtained from the filter residues were divided by the number of liters of water that had been filtered.
[2] Only DDT was found in suspended solids; the presence of DDD and DDE could not be confirmed in any case.

Insecticide Residues in Water and Sediment From Cisterns on the U.S. and British Virgin Islands—1970 [1]

Herbert Lenon[2], LaVerne Curry[2], Andrew Miller[2], and Daniel Patulski[2]

ABSTRACT

In the Virgin Islands the potential exists for pesticide contamination of water cisterns which supply and store all water used by local populations; cistern water and sediment samples on four islands were analyzed for pesticide residues in 1970 by gas-liquid chromatography. In the past, chlorinated hydrocarbon pesticides were used quite extensively on the Islands, however, malathion is more commonly used today.

Evidence of an unknown malathion metabolite was found in all 49 water samples analyzed, wereas malathion was found in only two (0.01 and 0.14 ppb). DDT, its metabolites, and dieldrin were not commonly found in the water samples except those from St. John where dieldrin was detected in approximately 50% of the samples (average concentration—0.19 ppb).

Sediment samples from cisterns, in general, contained much higher concentrations of pesticides than water, with DDT and its metabolites occurring most frequently. In many of these sediment samples, the residue levels were high enough to be concern. As a result it is strongly recommended that cisterns be cleaned frequently to remove sediment.

Introduction

Although the Virgin Islands receive an average annual rainfall of about 41 inches, much of this water is lost immediately by runoff and evaporation (*1*). Thus, the local populations depend on rain water for drinking and domestic use which is collected from roofs and stored in cisterns beneath their homes. With this type of rain catchment system, the nearby use of insecticides in mosquito eradication programs has been of concern, since insecticides could be carried by wind to roofs and then flushed into the cisterns during any of the Islands' frequent showers.

Cistern water supplies from four of the Virgin Islands were surveyed for insecticide accumulations. The cisterns sampled were on the three major U.S. Virgin Islands of St. Croix, St. Thomas, and St. John and one British Virgin Island, Anegada (Fig. 1). The Virgin Islands are at the end of a 1,300-mile chain of islands known as the Greater Antilles—including Cuba, Jamaica, and Puerto Rico—which begins off the southern tip of Florida and extends south and west toward Central America.

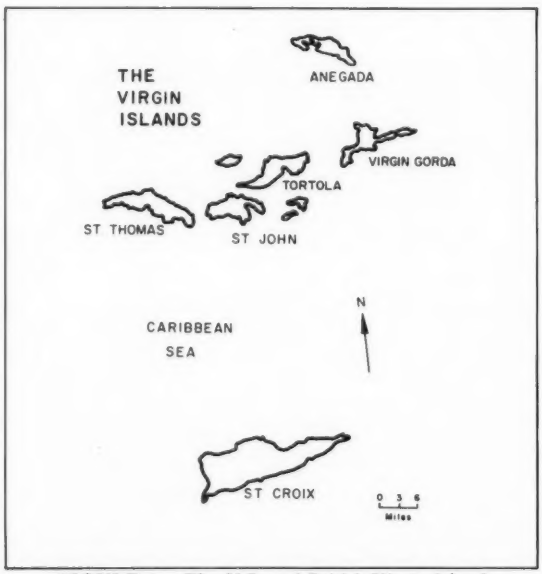

FIGURE 1.—*The U.S. and British Virgin Islands*

[1] Contribution 103 from the Virgin Islands Ecological Station. Great Lameshur Bay, St. John's, U.S. Virgin Islands.

[2] Department of Biology, Central Michigan University, Mt. Pleasant, Mich. 48858.

The U.S. Virgin Islands are of continental formation and were created during the Eocene Epoch by extrusion and folding from the ocean floor (6). St. John and St. Thomas are about 2 miles apart and are quite similar geographically. Both are rugged and hilly with numerous outcrops of metamorphic rocks. St. John is a small island, with 20 square miles of land, about two-thirds of which is national park. The 1960 population was 925 comprised mostly of natives who are supported largely by government funds. There is no commercial agriculture or significant tourism. Most of the population is centered around Cruz Bay and, to some extent, Coral Bay (Fig. 2). Small scattered native communities are also found on Bordeaux Mountain located in the interior south of Coral Bay. St. Thomas is larger and more densely populated than St. John, to the extent of being crowded, with 30 square miles of land and a population of 16,201 in 1960 (Fig. 3). For its economy, this island relies heavily on tourist spending which is approximately one million dollars a day. There is little industry and no major agriculture, although there is an Agricultural Experiment Station and some dairy farming.

St. Croix is 40 miles south of St. John. It has 85 square miles of surface area and had a population of 14,973 in 1960. Most of the population is centered around the two major cities of Frederiksted and Christiansted (Fig. 4). The economy is primarily industrial with rum as the main product, although there are a few remaining pineapple plantations. This island is also an important tourist center.

Anegada, one of the British Virgin Islands, is a flat coral island, almost entirely surrounded by reefs, 35 miles northeast of St. John and about 15 miles north of Virgin Gorda, the closest major island. The island is nearly 10 miles long and is almost divided by three large estuaries (Fig. 5). Nowhere is its elevation higher than 60 feet. It has the appearance of a large paved area and is exceedingly desolate. The population is very sparse, estimated at about 225 with only one small native community called "The Settlement." It is very poor with only shacks and junk accumulated for over 100 years in the front yards. The natives once fished for a

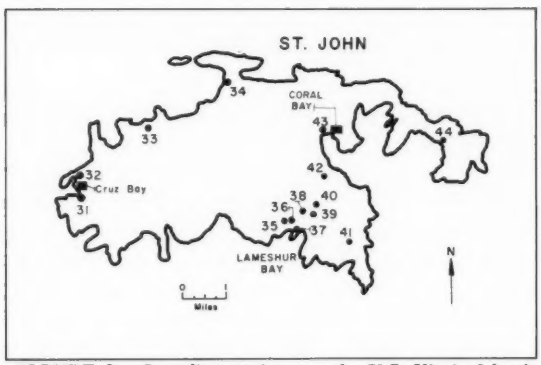

FIGURE 2.—*Sampling stations on the U.S. Virgin Island of St. John*

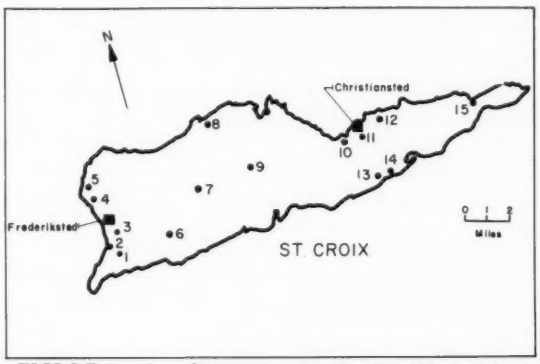

FIGURE 4.—*Sampling stations on the U.S. Virgin Island of St. Croix*

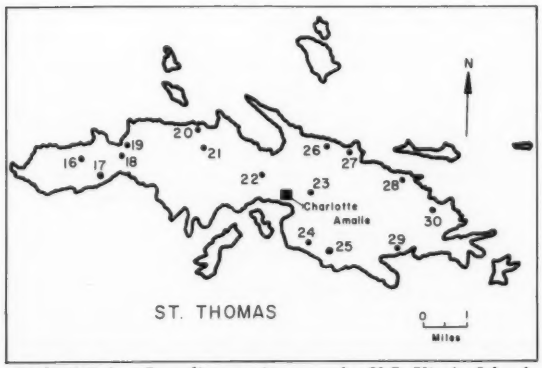

FIGURE 3.—*Sampling stations on the U.S. Virgin Island of St. Thomas*

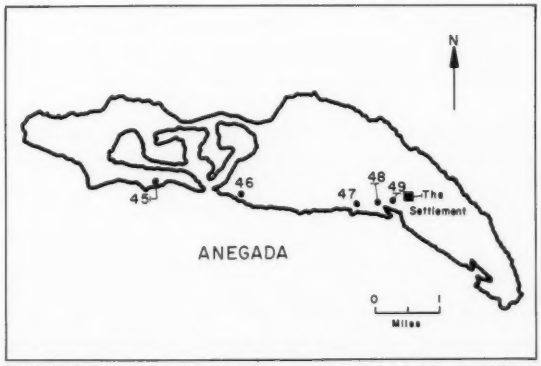

FIGURE 5.—*Sampling stations on the British Virgin Island of Anegada*

living but now work for a British corporation preparing for a jet airport. There is no tourism, agriculture, or industry.

Cisterns for catching rainwater are especially important for the basic supply of water since these islands have no other fresh water (*1*). The approximate average annual rainfalls for these four islands are: St. John, 47 inches; St. Thomas, 42 inches: St. Croix, 40 inches; and Anegada, 30 inches. Yet, these islands appear very dry, and as explained by Bowden *et al.* (*1*) "In essence rain falling on most rain-days is evaporated and transpired almost immediately, and is obviously of little or no consequence for growth of crops and vegetation."

Although no large-scale agricultural spraying is conducted on these islands, insecticides are used locally by individuals and by the U.S. Virgin Islands Health Department to control certain pest insects, especially mosquitoes. On St. Thomas and St. Croix, hotel owners spray areas to facilitate the tourist trade. The health department indicated that on St. Thomas DDT was used from 1960 to 1962 and dieldrin, from 1962 to 1964. Since then, malathion has been used, primarily because DDT and dieldrin were no longer effective against mosquitoes. According to Director Francois of Environmental Health (*personal communication, 1970*), it is applied to the ground, shrubbery, exposed water containers, and around the outside of water barrels and cisterns. Stored drinking water is often a source of mosquitoes, and private cisterns may be treated directly with Abate or DDVP resin strips. A similar program has been followed on St. Croix. On St. John, all insecticide use appears to be by individuals who apply much malathion. All insecticides are prohibited inside the national park.

Sampling Methods and Analytical Procedures

Samples of water and sediment from cisterns were taken on the following dates: St. Croix Island—March 18 and 19, 1970; St. Thomas Island—March 10-12, 1970; St. John Island—February 23 and March 9, 1970; and Anegada Island—March 26, 1970.

Each drinking water sample was collected in two 1-gal glass bottles. These were immediately extracted by liquid-liquid partitioning with purified petroleum ether using 70 ml per liter of water. This solvent was purified by twice distilling reagent grade petroleum ether with 10 g of dri-sodium per 3 liters at between 30° and 60° C. Each sample was extracted twice. Combined extracts were partially evaporated and sealed in screw-capped vials, packed, and sent by airmail to the biology laboratory at Central Michigan University, Mt. Pleasant, Mich. They were then immediately dried with anhydrous sodium sulfate, further evaporated, and adjusted to 25 ml for analysis. It is believed that little if any breakdown of the extracted pesticides occurred in the short period of transport as was also noted by Guerrant, Fetzer, and Miles (*2*) using hexane.

Sediment samples were collected with a plankton net attached to a metal pole from the bottoms of cisterns containing significant amounts of sediment. They were then sent in screw-capped bottles directly to Central Michigan University for extraction and analysis. Water in the samples was removed by filtering on Whatman No. 1 paper with a Buchner funnel. Then the paper containing the sediment was oven-dried overnight at 50° C. The sample (3-7 g) was weighed on the pre-weighed filter paper and then shaken thoroughly several times with purified petroleum ether. The sediment was washed three times in petroleum ether, then the entire extract was dried with anhydrous sodium sulfate, evaporated, and adjusted to 25 ml.

Sample extracts were identified and quantified by gas chromatography (Beckman GC-4) using electron capture detection. Column packing and operating parameters were based on those of Mills, Onley, and Gaither (*5*) and were very similar to those used later by Guerrant *et al.* (*2*) with malathion:

Column:	6′ x ⅛″ stainless steel, packed with 3% SE-30 on 60/80 mesh Chromosorb W
Temperatures:	Detector 300° C Column 180° C Inlet 210° C
Carrier gas:	Helium at a flow rate of 40 ml/min

Samples were verified by analysis using 3% OV-17 on Chromosorb W as described by Menzie and Prouty (*4*) and also used by Guerrant *et al.* (*2*) with good agreement of retention times between the two systems.

Laboratory tests gave the following average recoveries: DDE, 76.1%; TDE (DDD), 72.8%; DDT, 82.2%; dieldrin, 83.5%; and malathion, 85%. Data in this report have been corrected using these recovery rates.

The standards used were from:

A. Beckman Poly-Science Quant-Kits, 1% by weight in benzene, and included:
   - Technical *p,p′*-DDT, 99.5%
   - Technical *p,p′*-TDE, 99.5%
   - Technical dieldrin, 99.5%
   - Technical malathion, 99.5%

B. Pesticide Repository, Perrine Primate Laboratory, Environmental Protection Agency, prepared 1% by weight in petroleum ether:
   - Analytical Standard—*p,p′*-DDE, 99%
   - Analytical Standard—*o,p′*-TDE 99%
   - Analytical Standard—*o,p′*-DDT, 99%

A given quantity of malathion standard was allowed to hydrolyze naturally at room temperature for 6 months in order to characterize its metabolite in gas chromatographic analysis. This common metabolite was not identified but was used as a "qualitative standard."

Results and Discussion

DDT, its metabolites, and dieldrin were not commonly found in the water samples (Tables 1-4). DDT was not present in any of the water samples and its metabolites (DDE and TDE) were found in only one (Table 3). Dieldrin, which presents the greatest hazard in the water supply at levels found because of its toxicity and persistence, occurred in 48% (6 of 14) of the samples from St. John and 13% (2 of 15) of those from St. Thomas; however, no residues of dieldrin were found in samples from St. Croix and Anegada.

Malathion was present in only two water samples (Stations 19 and 34) and at low concentrations (0.14 and 0.01 ppb, respectively) (Tables 1-4). However, evidence of some metabolite of malathion was found in all water samples taken. Because this metabolite was not identified, values are not reported. Detection of this compound is reported only to suggest the previous presence of its precursor, malathion. Since malathion is a short-term insecticide, it was expected that it would be found only as one of its hydrolyzed products (*2*). Widespread use of this insecticide is, nevertheless, suggested through the common appearance of its metabolite in every water sample. The potential hazard of the insecticide would not appear to be great since hydrolysis occurs rather rapidly, especially under conditions of neutral-to-alkaline pH. Because the metabolite was not identified, however, the importance of its presence cannot be interpreted..

The highest indicated malathion occurrence, as evidenced by the greatest relative quantities of its metabolite, was on St. Thomas and St. John. It is reasonable to expect St. Thomas Island to have one of the highest levels of pesticide residues, since the local health department as well as individuals spray the area to reduce the annoyance of mosquitoes and the risk of malaria to both tourists and its large local population. It may be possible to explain the high occurrence of malathion in water samples from the cisterns of St. John on the basis of the large amounts of water for this island brought in from areas like Puerto Rico where insecticide use is probably more common. This would also perhaps, explain the more frequent occurrence of the chlorinated hydrocarbon pesticides, especially dieldrin (Table 3). Water samples from St. Croix were next highest in relative malathion metabolite, as might be expected, since it is also quite heavily populated and supports tourists interest.

TABLE 1.—*Stations from which cistern water was sampled on St. Croix Island*

Cistern Source	Approximate Age of Cistern (years)	Sample Station [1]
Residence	75-100	1
Business	9	2
Business	3-4	3
Hotel	>100	4
Residence	12	5
Museum	<10	6
Residence	13	7
School	10-15 (not in use)	8
Agric. Exp. Stn.	100 (plastic liner)	9
Hotel	5	10
Business	Unknown	11
Hotel	>100	12
Residence	Unknown	13
Residence	1	14
Hotel	Unknown (cleaned often)	15

NOTE: No residues of DDT, its metabolites, dieldrin, or malathion were detected. Evidence of some metabolite of malathion was found in all water samples taken.

[1] Sample station numbers correspond with those shown on map (Fig. 4).

TABLE 2.—*Stations from which cistern water was sampled on St. Thomas Island showing pesticide residue levels detected*

Cistern Source	Approximate Age of Cistern (years)	Sample Station [1]	Residues in ppb	
			Malathion	Dieldrin
Estate	>100	16	—	—
Estate	5	17	—	—
Estate	30	18	—	—
Residence	>30	19	0.14	0.04
Dept. of Agric.	>30 (artesian)	20	—	—
Nursery	39	21	—	—
Public	4 (open)	22	—	—
Hospital	40 (open)	23	—	0.10
Residence	3	24	—	—
Residence	200	25	—	—
Camp (Peace Corp)	>30	27	—	—
Residence	5	28	—	—
Race track	1	29a	—	—
Race track	21	29b	—	—
School	>30	30	—	—

NOTE: — = no residue detected; no residues of DDT or its metabolites were detected. Evidence of some metabolite of malathion was found in all water samples taken.

[1] Sample station numbers correspond with those shown on map (Fig. 3).

Sediment was found in cisterns and consequently sampled at 35 of the 46 stations where water samples were obtained. Pesticide residues were detected in sediment samples from only 15 of the Stations (Table 5). Generally sediment samples had much higher residues with more variation in levels than water samples (Table 5—values for sediment were reported in ppm rather than in ppb). This suggests that when sediment occurs, the insecticides tend to be bound and accumulate over longer periods of time. Of the pesticides detected, DDT, its metabolites, or both occurred most frequently and were present in 4 of 8 sediment samples from St. John and 9 of 14 from St. Thomas. The relative absence of these compounds in both sediment and water samples from St. Croix and Anegada indicates little usage of the pesticides on these islands. Many of the residue levels of DDT and its metabolites in sediment samples from St. John and St. Thomas were high enough to be of considerable concern, particularly two from Stations 19 and 41 (Table 5). It appears that DDT was used in these two situations in place of malathion because malathion was not available. Sediment from Station 41 contained a high amount of DDT and smaller amounts of its degradation products indicating a short-term occurrence of the pesticide in the sediment, while sediment from Station 19 contained very high amounts of metabolites and little DDT, thus, indicating a longer presence of the pesticide. The fact that unusually high amounts were found only at these two locations suggests individual sprayings and, perhaps, careless application.

Dieldrin was not found in sediment as frequently (only one sample) as in water, a finding which remains unexplained. Malathion was found in only one sediment sample, while its metabolite was found in all but five (Stations 19, 22, 32, 41, and 43). It is interesting to note that even though evidence of this product was found in all water samples, it was absent in five of the sediment samples.

Most sediment samples were composed of silty loam or organic matter and sometimes coral sand. DDT and its degradation products can accumulate in sediment, since they are bound to solid particles. Lichtenstein and Schulz (*3*) demonstrated that the persistence of residues in soils is dependent on various factors such as the insecticide itself, soil types, as well as climatic conditions. Soils of higher organic content tend to bind the greatest amount of chlorinated hydrocarbon pesticide.

The survey and evaluation of pesticide levels in private and public cisterns on these islands were complicated for several reasons. The exact sources of the water are never known. Although rainwater is collected in all cisterns, these supplies are frequently insufficient and additional water must be purchased from various places, often as far away as Puerto Rico, but including other islands as well. This is known to be true, at least, for St. John where water arrives by barge and is then

TABLE 3.—*Stations from which cistern water was sampled on St. John Island showing pesticide residue levels detected*

Cistern Source [1]	Sample Station [2]	Residues in ppb				
		Malathion	DDT	TDE	DDE	Dieldrin
Public	31	—	—	—	—	—
National park	32	—	—	—	—	—
Camp ground	33	—	—	—	—	—
Residence	34	0.01	—	—	—	0.02
Old commissary	36	—	—	—	—	0.01
Laboratory	37	—	—	—	—	—
Residence (Park Ranger)	38	—	—	—	—	0.04
Camp	39a	—	—	—	—	—
Camp	39b	—	—	—	—	—
Laboratory	40	—	—	—	—	1.03
Residence	41	—	—	0.15	0.02	—
Clinic	42	—	—	—	—	0.04
Church	43	—	—	—	—	0.01
School	44	—	—	—	—	—
Average						0.19

NOTE: — = no residue detected. Evidence of some metabolite of malathion was found in all water samples taken.

[1] Approximate ages of these cisterns are unknown.

[2] Sample station numbers correspond with those shown on map (Fig. 2).

TABLE 4.—*Stations from which cistern water was sampled on Anegada Island*

Cistern Source	Approximate Age of Cistern (years)	Sample Station [1]
Business	<1 (open)	45
Residence	<1	46
Public well	(plastic liner) very old	47
Public well	Unknown	48
School	>25	49

NOTE: No residues of DDT, its metabolites, dieldrin, or malathion were detected. Evidence of some metabolite of malathion was found in all water samples taken.

[1] Sample station numbers correspond with those shown on map (Fig. 5).

trucked around the Island. Consequently, this may account for the occurrence in water of pesticides not used locally. Incomplete information was also a problem. Data regarding the age and characteristics of the individual water supplies were often sparse. Some cisterns were cleaned regularly and contained no sediment, while others were never cleaned and contained several inches of bottom material.

No apparent correlations are evident between the age of cisterns and residue levels, or between commercial or residential establishments and residue levels. It appears to be more a matter of the extent of individual use of pesticides, care in application, as well as, perhaps, the location where water was obtained.

Results do not suggest a potential danger to the people who use water from the cisterns examined with the possible exception of some on St. John containing significant levels of dieldrin. Sediment, contained in many of the cisterns, represents a much greater potential hazard with accumulated levels of residues, representing the past presence of insecticides at various stages of degradation. It is suggested that these sediment accumulations be removed by frequent cleaning of the cisterns, thus keeping total residue levels in the cisterns to a minimum.

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See Appendix for the chemical names of compounds discussed in this paper.

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TABLE 5.—*Stations where sediment of cisterns was sampled and pesticide residue concentrations detected*

Sample Station [1]	Residues in ppm				
	Malathion	DDT	TDE	DDE	Dieldrin
ST. CROIX					
3	—	—	—	—	—
4	—	—	—	—	—
5	—	—	—	—	—
6	—	—	—	—	—
8	—	—	—	—	—
9	—	—	—	—	—
10	—	—	—	—	—
12	—	—	—	—	—
15	—	—	—	—	—
ST. THOMAS					
16	—	0.61	0.37	0.09	—
18	0.19	37.49	11.50	0.96	—
19	—	16.36	401.37	1,250.26	—
20	—	13.49	4.12	1.58	—
22	—	—	—	—	—
23	—	—	—	—	—
24	—	—	—	—	—
25	—	—	—	—	—
26	—	—	—	—	—
27	—	0.96	0.43	0.17	—
28	—	—	—	0.07	—
29a	—	—	0.66	0.89	—
29b	—	0.28	0.22	0.32	—
30	—	0.09	0.10	0.12	—
ST. JOHN					
32	—	—	—	—	—
33	—	—	—	—	—
34	—	—	—	—	—
35	—	1.82	0.21	0.05	—
36	—	—	—	—	0.08
41	—	271.29	34.11	6.27	—
43	—	—	—	0.14	—
44	—	2.77	0.44	0.07	—
ANEGADA					
45	—	—	—	—	—
47	—	—	—	—	—
48	—	—	—	—	—
49	—	7.18	1.79	3.68	—

NOTE: — = no residue detected; evidence of some metabolite of malathion was found in all sediment samples with the exception of the five samples from the following stations: Stations 19, 22, 32, 41, and 43.

[1] Sample station numbers correspond to those for water samples (Tables 1-4; Figs. 2-5).

PESTICIDES IN SOIL

Pesticide Residue Levels in Soils, FY 1969—National Soils Monitoring Program

G. B. Wiersma[1], H. Tai[2], and P. F. Sand[3]

ABSTRACT

This report is a summary of the FY 1969 results of the National Soils Monitoring Program, an integral part of the National Pesticide Monitoring Program (NPMP). Pesticide residues in cropland soil for 43 States and noncropland soil for 11 States are reported. Tables for each State give the number of samples collected, arithmetic means and ranges of residue levels detected, and the percent of sites with detectable residues. In addition, for selected pesticides and various States and State groupings, a frequency distribution of pesticide residues was determined. Use records for FY 1969 are given by the pesticides used, the percent of sites treated, the average application rates, and the average amounts applied per site. Comparisons are made between residue levels in different land-use areas.

Introduction

The National Soils Monitoring Program is an integral part of the National Pesticide Monitoring Program (NPMP), which was initiated as a result of a recommendation made by the President's Science Advisory Committee in its report of 1964 entitled "Use of Pesticides" that the appropriate Federal agencies "develop a continuing network to monitor residue levels in air, water, soil, man, wildlife, and fish." The NPMP as originally designed was described in the first issue of the *Pesticides Monitoring Journal* (*1*), and a revised description to reflect certain program realignments and other changes was published in the June 1971 issue of this *Journal* (*2*).

The objectives of the NPMP are to determine levels and trends of pesticides in the various components of the environment (*2*). The establishment of baseline or background levels of pesticide residues through the NPMP will provide a basis for comparison of subsequently identified pesticide residue levels in an environmental component.

The Panel on Pesticides Monitoring of the Working Group on Pesticides (*2*) listed five bases for concern to be used in evaluating pesticide residue levels in the various environmental components. They are:

(1) any concentration of a pesticide known to be potentially harmful;
(2) increasing trends;
(3) exceeding standards;
(4) recognition of adverse effects on humans; and
(5) erratic variability (a statistically oriented observation that is potentially common to each stratum sampled).

The results of this study serve to establish a baseline of pesticide residues in cropland and noncropland soils at a particular point in time (FY 1969). The present data and all future data will be evaluated using applicable criteria included in the five bases of concern outlined above.

Sampling Procedures and Methods

In general, sampling techniques involved in this study were the same as those described by Wiersma, Sand, and Cox (*3*).

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In FY 1969, cropland soil was sampled in every State except Alaska, Hawaii, Kansas, Minnesota, Montana, Oregon, and Texas. Noncropland was sampled in 11 States—Arizona, Georgia, Idaho, Iowa, Maine, Maryland, Nebraska, Virginia, Washington, West Virginia, and Wyoming. Samples collected in FY 1969 included both soil and mature crops and/or those ready for harvest; however, results of crop analyses are not published in this report.

Analytical Procedures

ORGANOCHLORINE AND ORGANOPHOSPHOROUS COMPOUNDS

A subsample of soil weighing 300 g, wet weight, was placed in a 2-qt fruit jar with 600 ml of 3:1 hexane-isopropanol solvent. The jars were sealed and rotated for 4 hours. After rotation, the soil was allowed to settle, and 200 ml of the extract solution was filtered into a 500-ml separatory funnel. Isorpropanol was removed with two washings of distilled water, and the remaining solution was then filtered through a funnel containing glass wool and anhydrous sodium sulfate (Na_2SO_4). Further cleanup was normally not required before analysis.

Gas-Liquid Chromatography

Analyses were performed on gas chromatographs equipped with tritium foil electron affinity detectors for organochlorine compounds and thermionic or flame photometric detectors for organophosphorous compounds. A dual-column system employing polar and nonpolar columns was utilized to identify and confirm pesticides. Instrument parameters were as follows:

Columns:	Glass, 183 cm long by 6 mm, o.d., and 4 mm, i.d., with one of the following packings: 3% DC-200 on 100/120 mesh Gas Chrom Q or 9% QF-1 on 100/120 mesh Gas Chrom Q
Carrier gas:	5% methane-argon at a flow rate of 80 ml/min
Temperatures:	Detector 200° C Injection port 250° C Column QF-1 166° C Column DC-200 170°-175° C

When necessary, confirmation of residues was made by thin layer chromatography or p-values. The lower limit of detection was 0.01 ppm. The average recovery rate for all pesticides was 100% (with a ±10% error); the data were corrected for recovery and also adjusted to a dry-weight basis by determining the moisture content on a separate portion of each sample using the oven drying method.

ATRAZINE

After a 4-hour Soxhlet extraction of a 50-g subsample of soil with 25 ml of water and 300 ml of methanol, the sample extract was transferred to a 1-liter separatory funnel and 200 ml of water added. The sample extract was partitioned three times with a portion of 150 ml of freon 113 for each partitioning. The freon 113 fractions were combined and concentrated to incipient dryness. The sample was then dissolved in hexane, adjusted to a 5-ml volume, and injected into a gas-liquid chromatograph.

Gas-Liquid Chromatography

A thermionic flame detector with rubidium sulfate coating on a helix coil was used. Instrument parameters were as follows:

Column:	Glass, 183 cm long by 6 mm, o.d., and 4 mm, i.d., packed with 3% Versamid 900 on 100/120 mesh Gas Chrom Q
Carrier gas:	Helium
Detector fuel gas:	Oxygen (200-300 ml/min); Hydrogen (20-30 ml/min)
Temperatures:	Detector 240° C Injection port 240° C Column 240° C

Confirmation was made using a DC-200 column at 180° C and a Coulson detector (reductive mode) at the following temperature settings: pyrolysis tube—850° C, transfer line—220° C, and block—220° C.

The minimum detection limit was 0.01 ppm, and recovery was about 100%.

2,4-D

Analyses were made following the procedure developed by Woodham *et al.* (*4*). The analytical method involved a diethyl ether extraction of acidified soil, an alkali wash to remove interfering substances, and an esterification procedure using 10% boron trichloride in 2-chloroethanol reagent. The 2-chloroethyl ester of 2,4-D was then analyzed by gas chromatography. The minimum detection limit was 0.01 ppm, and the average recovery was 85%. Results were corrected for percent recovery.

ARSENIC

Arsenic was determined by atomic absorption spectrophotometry. The soil sample was first extracted with 9.6N hydrochloric acid (HCL) and reduced to trivalent arsenic with stannous chloride. The trivalent arsenic was partitioned from HCL solution to benzene, then further partitioned into water for the absorption measurement. A Perkin-Elmer Model 303 instrument was used, and absorbance was measured with an arsenic lamp at 1972 Å with argon as an aspirant to an air-hydrogen flame. The minimum detection limit was 0.1 ppm, and the recovery value for arsenic averaged 70%. Results were corrected for percent recovery.

Results

The data in this report are for soils only (both cropland and noncropland) and include results for all States

sampled in the study. Caution should be exercised when interpreting the arithmetic means presented in the tables, because pesticide residue data are not normally distributed, and the arithmetic means for pesticide residues tend to be greater than the corresponding median. Therefore, they cannot be considered an indication of the central tendency of the data. Information accompanying the arithmetic means in this report such as the percent occurrence, range of detected residues, and number of observations can aid in evaluating the arithmetic mean.

RESIDUES—ALL STATES

Table 1 presents a summary of pesticide residues in cropland soils for all 43 States sampled. Percent occur-

TABLE 1.—*Summary of pesticide residues in cropland soil from 43 States—FY 1969*

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
Aldrin	1,729	189	10.9	0.02	0.01-3.06
Arsenic	1,726	1,713	99.3	6.43	0.25-107.45
Atrazine	199	28	14.1	0.01	0.01-1.55
Carbophenothion	66	1	1.5	<0.01	0.23
Chlordane	1,729	151	8.7	0.04	0.01-6.30
2,4-D	188	3	1.6	<0.01	0.01-0.03
DCPA (Dacthal®)	1,729	1	0.1	<0.01	0.54
o,p'-DDE	1,729	79	4.6	<0.01	0.01-0.20
p,p'-DDE	1,729	429	24.8	0.06	0.01-6.99
o,p'-DDT	1,729	243	14.1	0.03	0.01-6.29
p,p'-DDT	1,729	384	22.2	0.17	0.01-35.92
DDTR	1,729	451	26.1	0.31	0.01-78.36
DEF	1,729	1	0.1	<0.01	0.12
Diazinon	66	2	3.0	<0.01	0.02-0.15
Dicofol	1,729	9	0.5	<0.01	0.03-1.07
Dieldrin	1,729	480	27.8	0.03	0.01-1.60
Endosulfan (I)	1,729	5	0.3	<0.01	0.01-0.24
Endosulfan (II)	1,729	9	0.5	<0.01	0.01-0.53
Endosulfan sulfate	1,729	11	0.6	<0.01	0.01-0.94
Endrin	1,729	39	2.3	<0.01	0.01-0.56
Endrin aldehyde	1,729	1	0.1	<0.01	0.03
Endrin ketone	1,729	9	0.5	<0.01	0.01-0.13
Ethion	66	1	1.5	<0.01	0.03
Heptachlor	1,729	68	3.9	<0.01	0.01-0.97
Heptachlor epoxide	1,729	139	8.0	<0.01	0.01-1.08
Isodrin	1,729	11	0.6	<0.01	0.01-0.03
Lindane	1,729	15	0.9	<0.01	0.01-0.35
Malathion	66	2	3.0	0.01	0.04-0.36
Methoxychlor	1,729	1	0.1	<0.01	0.28
Ethyl parathion	66	7	10.6	0.06	0.01-3.01
PCNB	1,729	1	0.1	<0.01	0.69
o,p'-TDE	1,729	49	2.8	0.01	0.01-4.52
p,p'-TDE	1,729	265	15.3	0.05	0.01-31.43
Toxaphene	1,729	73	4.2	0.07	0.10-11.72
Trifluralin	1,729	60	3.5	<0.01	0.01-0.25

[1] One sample per site.
[2] Percent based on number of sites with residues greater than or equal to the sensitivity limits.

rence of residues is based on the number of sites with residues greater than or equal to the sensitivity limit.

The data for atrazine, 2,4-D, and the organophosphates are not truly comparable with those determined for the organochlorines or arsenic, because analyses for atrazine and 2,4-D were made only when use records indicated that they had been applied—199 and 188 times, respectively, and analyses for organophosphates were performed on only 66 of the 1,729 samples.

Elemental arsenic residues were found most frequently, with 99.3% of the sites having detectable residues and a mean level of 6.4 ppm. It is probable that most of this arsenic was from natural sources, although agricultural sources cannot be ruled out at this time.

The most widely distributed organochlorine pesticide was dieldrin, with 27.8% of the sites having detectable residues, followed by DDTR residues (a compilation of all members of the DDT group) found at 26.1% of the sites; aldrin, found at 10.9%; and chlordane, found at 8.7%. DDTR had the highest mean residue level, with 0.31 ppm found in cropland soils. With the exception of individual members of the DDT group, the other organochlorines had average residues ranging from <0.01 to 0.07 ppm.

Based on the 66 samples analyzed for organophosphates, ethyl parathion was detected 10.6% of the time, with a mean residue level of 0.06 ppm. Malathion and diazinon were each detected 3.0% of the time, with mean residue levels of 0.01 and <0.01 ppm, respectively.

In the 188 samples analyzed for 2,4-D and other chlorophenoxy herbicides, 2,4-D was the only one detected; 2,4-D was found in 1.6% of 188 samples analyzed, with a mean residue level of <0.01 ppm. Atrazine was detected in 14.1% of the 199 samples analyzed, with a mean residue level of 0.01 ppm—the highest mean residue of the herbicides detected. Trifluralin was detected in 3.5% of the 1,729 samples, with a mean residue level of <0.01 ppm.

The residues found in noncropland soils for the 11 States sampled are presented in Table 2. The mean arsenic residue level was 5.0 ppm, occurring in 98.5% of the samples. DDTR was detected in 16.1% of the noncropland soils at levels ranging from 0.01 to 0.62 ppm, with a mean level of 0.01 ppm. With the exception of members of the DDT group, dieldrin was the most widely distributed pesticide, occurring in 4.0% of the samples, with residues ranging between 0.01 to 0.09 ppm and a mean residue level of <0.01 ppm.

RESIDUES—INDIVIDUAL STATES

The pesticide residue summaries for cropland by individual States are given in Table 3, and similar results are shown for noncropland in Table 4. It would be impractical to attempt to comment on the results for each State; therefore, in order to facilitate summarizing the data, Figs. 1, 2, and 3 are presented. These are for three of the most commonly occurring residues—arsenic, DDTR, and dieldrin. Means for each pesticide in each State were calculated, and distribution of these averages are indicated on the corresponding Figures.

TABLE 2.—*Summary of pesticide residues in noncropland soil from 11 States—FY 1969*

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
Aldrin	199	1	0.5	<0.01	0.02
Arsenic	198	195	98.5	5.01	0.33-54.17
Chlordane	199	3	1.5	<0.01	0.04-0.50
o,p'-DDE	199	1	0.5	<0.01	0.02
p,p'-DDE	199	27	13.6	0.01	0.01-0.31
o,p'-DDT	199	7	3.5	<0.01	0.01-0.05
p,p'-DDT	199	18	9.1	0.01	0.01-0.23
DDTR	199	32	16.1	0.01	0.01-0.62
Dicofol	199	2	1.0	<0.01	0.10-0.29
Dieldrin	199	8	4.0	<0.01	0.01-0.09
Heptachlor epoxide	199	2	1.0	<0.01	0.01
p,p'-TDE	199	6	3.0	<0.01	0.01-0.18
Toxaphene	199	1	0.5	<0.01	0.52

[1] One sample per site.
[2] Percent based on number of sites with residues greater than or equal to the sensitivity limits.

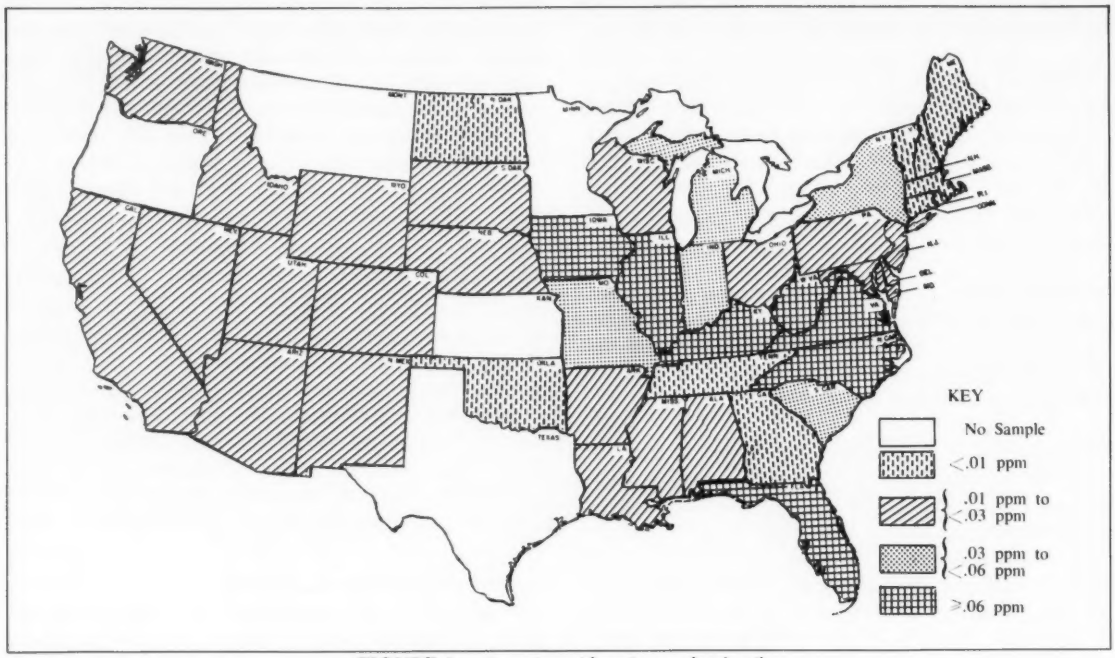

FIGURE 1.—*Arsenic residues in cropland soil*

The class intervals for the keys accompanying each Figure were obtained in the following manner: The range of residues for the Nation was obtained, and the highest value was converted to a logarithm. This value was then divided by the number of desired classes. The resulting intervals were added to obtain the class boundaries which, in turn, were converted to the untransformed dimensions. Essentially, this took advantage of the fact that most residue data are logarithmically distributed.

Distribution of arsenic residues across the United States is presented in Fig. 1. The highest residue levels were found in the New England States (Connecticut, Maine, Massachusetts, New Hampshire, Rhode Island, and Vermont), Arkansas, Kentucky, New York, North Dakota, Ohio, and Pennsylvania; these individual States and the New England States had mean residues of arsenic >8.4 ppm. The remaining residues were distributed primarily in the 2.0 to 8.4 ppm range, with Wyoming and Florida having less than 2.0 ppm. Those States left blank were not sampled.

The distribution of DDT residues (DDTR) is shown in Fig. 2. Once again, the key indicates the range of residues for each of the class intervals. A similar map for dieldrin residues is presented in Fig. 3.

The mean residue levels, the percent positive sites, and the range of residue levels for the 12 States with the highest arsenic residues are shown in Table 5.

Residue data for the five States with the highest DDTR residues are presented in Table 6. Although Michigan had a mean residue of 2.09 ppm and a range of 0.01 to 78.36 ppm, only 23.5% of the samples had detectable residues, indicating that the residues were not widely distributed. By contrast, Mississippi had a mean residue of 2.06 ppm with 89.7% of its sites having detectable residues and a narrower range (0.03 to 13.14 ppm). Although the range was narrower, pesticide residues were more widely distributed in Mississippi than in Michigan.

The seven States wth the highest dieldrin residues are listed in Table 7. The highest mean residue level, 0.11 ppm, was found in Illinois, with 61.3% of the sites having detectable residues. In general, the other six States tended to have mean residues approximating one another, 0.06, 0.07, or 0.08 ppm.

PESTICIDE USE RECORDS

When soil samples were collected, an attempt was made to determine what pesticides had been used on the sites for the year of sampling. The summary tables for the use records show the percent of times a pesticide was

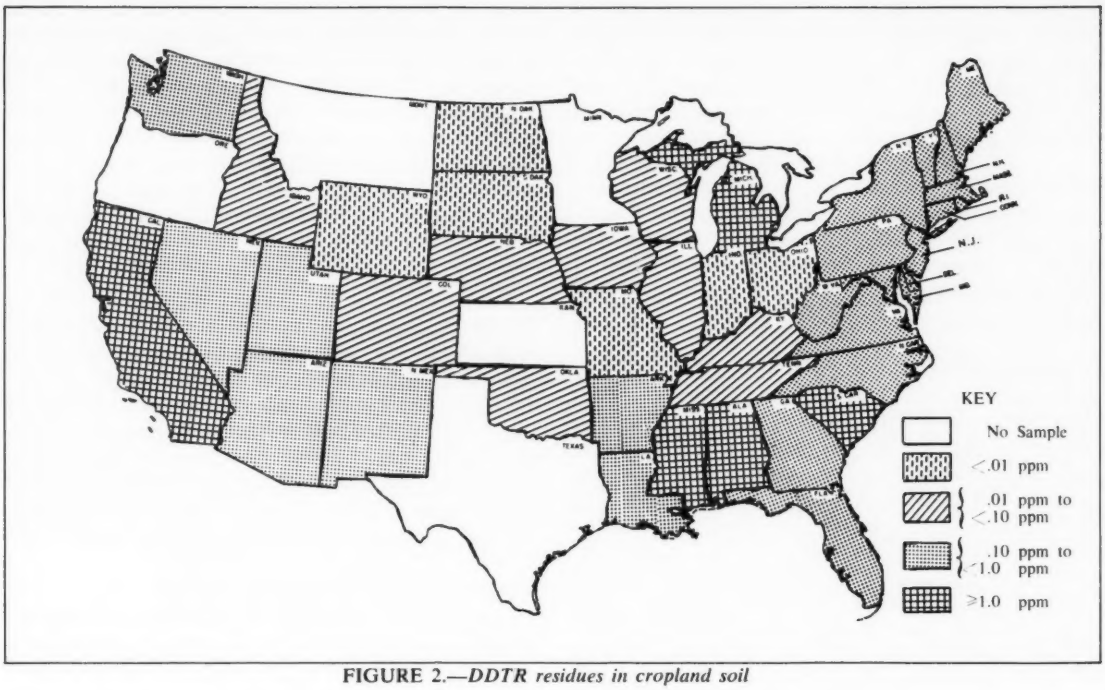

FIGURE 2.—*DDTR residues in cropland soil*

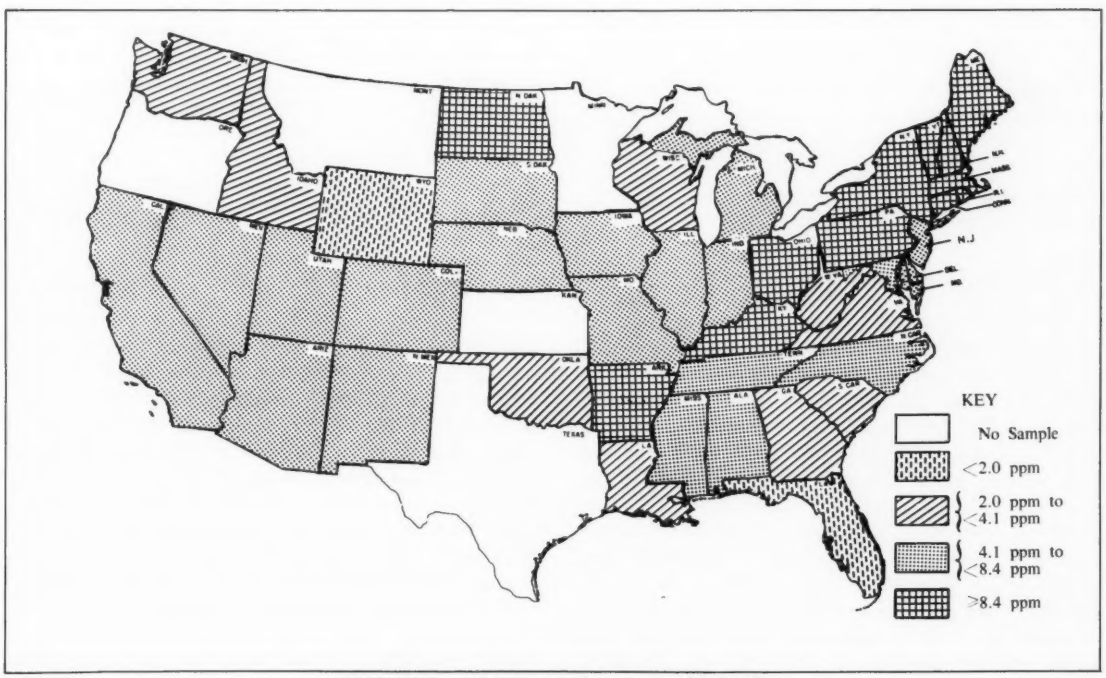

FIGURE 3.—*Dieldrin residues in cropland soil*

used, the average application rate expressed in pounds per acre of the active ingredients, and the average amount applied per site. The average amount per site was determined by dividing the total amount of active ingredient of a pesticide used by the total number of sites surveyed.

Table 8 shows 130 different pesticides reported to have been used on cropland in the year of sampling. Those most commonly used were atrazine, captan, 2,4-D, malathion, and methylmercury dicyandiamide. Technical DDT was used on 3.44% of the sites, aldrin on 4.16% of the sites, and dieldrin on 1.19% of the sites.

On noncropland sites 2,4-D, malathion, and mirex were reported to have been used (Table 9). However, these should not be considered the only pesticides used on noncropland sites. In general, records of treatment of noncropland sites are less accurate than those kept for cropland. The breakdown of pesticide usage by individual States for cropland and noncropland soils, respectively, are shown in Tables 10 and 11. Of the 43 States with cropland soil analyzed, use records for 4 showed no pesticides used on the sampling sites: Nevada (2 sites); New Hampshire (2 sites); Vermont (5 sites); and Wyoming (17 sites). Of the 11 States with noncropland soil analyzed, 8 reported no pesticides used on the sampling sites: Arizona (43 sites); Iowa (7 sites); Maine (11 sites); Maryland (3 sites); Virginia (14 sites); Washington (11 sites); West Virginia (9 sites); and Wyoming (37 sites).

Because of the number of States and pesticides presented in Tables 10 and 11, it is difficult to make all possible comparisons between the use patterns indicated and the detected residues shown in Tables 3 and 4. Therefore, comparisons have been restricted to those States having the highest residues as shown in Figs. 1, 2, and 3 (arsenic, DDTR, and dieldrin, respectively).

Table 12 compares those States having the highest arsenic residues with the average amount applied per site and the percent of sites which reported using an arsenic compound. The amount of arsenic applied did not seem to be directly related to the amount detected in the soil. Arkansas, Kentucky, North Dakota, and Ohio reportedly used no arsenic compounds, whereas New England, New York, and Pennsylvania reported using sodium arsenite and lead arsenate. The application rates were below the detected residue levels, and the percent of times used was below the percent of times residues were detected. It also must be considered that the application rates were for the active ingredients of sodium arsenite and lead arsenate, and not for elemental arsenic alone. A fair assumption would be that most arsenic residues detected in cropland soils probably resulted from natural levels of arsenic.

A similar comparison for the five States with the highest DDTR residues is found in Table 13. It is interesting to note that use records for four of the States listed (California, Michigan, Mississippi, and South Carolina) indicate that the amount applied was less than the mean level detected in the soil. Also, in all five States, the percent of sites positive for DDTR was approximately three or four times greater than the percent of sites reportedly treated with DDT. Unlike arsenic, the residues of DDTR could only result from the use of DDT either in the year of sampling or in previous years.

Table 14 lists the seven States with the highest dieldrin residues. In most cases, the average amount of aldrin/dieldrin applied approximated the mean residue of dieldrin detected in the soil, but the percent of sites reportedly treated with dieldrin or aldrin was always consideably less than the percent of sites with dieldrin residues. This wider distribution of dieldrin residues, when compared to use records for the year of sampling, probably indicates residues from previous years.

PESTICIDE FREQUENCY DISTRIBUTION

The statistics discussed thus far, namely the mean, the range, and the percent of sites at which residues were detected, do not describe their distribution. To describe this distribution, probit analysis was used. The residue levels were ranked from lowest to highest, accumulated, and the percentages computed. The residues were transformed to logarithms, the percentages to probits, and the relationship between the logarithms of the residues and the probits of the accumulated percentages was calculated by regression analysis. The computer program used was that of Daum. (*5*); the theory and techniques as applied in the cited reference were modified slightly.

The residue levels at the fiftieth percentile point (median) for the individual pesticides in soil for each State along with the upper and lower 95% fiducial limits are presented in Table 15. For example, in the State of Alabama, the fiftieth percentile point (median) for arsenic was 4.09 ppm. Thus, 50% of the sites had residues less than 4.09 ppm. The upper and the lower fiducial limits of the residues establish the 95% confidence interval about the residue value for the fiftieth percentile. It should be noted that the mean for a particular State is not the same as the fiftieth percentile point (median) from the frequency distribution. For example, the mean level of arsenic for Alabama was 6.1 ppm, while the frequency distribution indicated 4.09 ppm for the fiftieth percentile point. This is an example of the fact that residue data are not normally distributed and the mean and median are not identical.

Not all pesticides are shown for all States. A cutoff point of six or more pairs of observations was used to eliminate

situations where there were too few observations to calculate a reliable distribution. Space did not permit printing tables showing distribution of pesticide residues for percentiles other than the fiftieth.

CROPPING REGIONS ANALYSIS

The data were grouped by counties into various cropping regions, and these are shown in Tables 16 and 17. The boundaries for the various cropping areas were based on a major land-use map of the United States compiled by F. J. Marschner of the U.S. Department of Agriculture, Bureau of Agricultural Economics, 1950. No effort was made to make a land-use division within counties. This resulted in a good definition of the larger land-use areas such as the corn belt and cotton-growing areas. The land in the United States was grouped into several major land-use areas—corn, cotton, general farming, hay, small grain, vegetables, and fruit. In some cases, two areas overlapped. Irrigated land was determined from information obtained at the time of sample collection in this study.

It is of interest to make a few individual comparisons between the cropping regions and the national means. For example, note that in the corn region, aldrin occurred 23.5% of the time (Table 17) with a mean residue level of 0.05 ppm (Table 16). However, nationally, aldrin only occurred 10.9% of the time with a mean level of 0.02 ppm (Table 1), an indication of the heavier use of aldrin in the corn region. But, in the corn region, the mean residue level of DDTR was 0.14 ppm which is well below the national mean of 0.31 ppm.

The vegetable and fruit cropping region had the highest level of DDTR, over two times higher than the next highest cropping region and over six times higher than the national mean for DDTR. This might result from a high use of DDT in various orchard operations. The next highest residue was found in the cotton and vegetable region, with approximately equal amounts detected between them. The rest of the amounts of DDT in the cotton and general farming, general farming, hay and general farming, and irrigated land were similar to one another. The two areas with the least amount of DDTR in the soil were the corn and small grains cropping regions.

The corn, vegetable, and vegetable and fruit cropping regions had the heaviest residues of dieldrin. Residues of dieldrin in the other cropping regions were either equal to or below the mean residues detected for all States (Table 1).

The cotton cropping region had the highest toxaphene residues. The cotton and general farming and general farming cropping regions had residue levels of about half those detected in the cotton cropping region.

Acknowledgment

It is not possible to list, by name, all the people who contributed to this study; however, special mention is made of the staff at the Monitoring Laboratory, Mississippi Test Facility, Bay St. Louis, Miss., who processed and analyzed the samples for chemical residues and contributed immeasurably to this study and of the inspectors from the Animal Plant Health Inspection Service (APHIS) who collected the samples. Finally, recognition is due Dr. Edwin Cox, Biometrical Services Staff, USDA, for the sample allocation procedures and to Dr. Richard Daum of the Animal Plant Health Inspection Service, USDA, for the probit analyses.

See Appendix for chemical names and compounds discussed in this paper.

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TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
ALABAMA					
Arsenic	23	23	100.0	6.11	0.70-28.60
Chlordane	22	3	13.6	0.04	0.07-0.62
o,p'-DDE	22	1	4.6	<0.01	0.01
p,p'-DDE	22	19	86.4	0.17	0.01-0.72
o,p'-DDT	22	16	72.7	0.09	0.01-0.65
p,p'-DDT	22	20	90.9	0.78	0.02-6.60
DDTR	22	20	90.9	1.13	0.05-8.08
Dieldrin	22	5	22.7	0.01	0.01-0.14
Endrin	22	2	9.1	<0.01	0.03-0.05
Heptachlor	22	2	9.1	<0.01	0.01
Heptachlor epoxide	22	3	13.6	<0.01	0.01-0.04
Lindane	22	2	9.1	<0.01	0.01
o,p'-TDE	22	1	4.6	<0.01	0.08
p,p'-TDE	22	13	59.1	0.07	0.01-0.73
Toxaphene	22	6	27.3	0.69	0.68-4.95
Trifluralin	22	7	31.8	0.01	0.01-0.08
ARIZONA					
Arsenic	8	8	100.0	6.58	2.82-9.97
o,p'-DDE	8	4	50.0	0.02	0.01-0.07
p,p'-DDE	8	8	100.0	0.46	0.06-0.84
o,p'-DDT	8	5	62.5	0.07	0.08-0.17
p,p'-DDT	8	7	87.5	0.20	0.03-0.57
DDTR	8	8	100.0	0.76	0.06-1.56
Endosulfan (I)	8	1	12.5	0.03	0.24
Endosulfan (II)	8	1	12.5	0.07	0.53
Endosulfan sulfate	8	1	12.5	0.04	0.29
Endrin	8	3	37.5	0.07	0.10-0.22
Endrin ketone	8	2	25.0	0.01	0.01-0.07
p,p'-TDE	8	2	25.0	0.01	0.03-0.06
Toxaphene	8	6	75.0	1.09	0.57-4.27
Trifluralin	8	1	12.5	0.02	0.13
ARKANSAS					
Aldrin	47	7	14.9	<0.01	0.01-0.06
Arsenic	47	47	100.0	8.98	1.70-28.25
o,p'-DDE	47	5	10.6	0.01	0.01-0.07
p,p'-DDE	47	32	68.1	0.24	0.01-2.81
o,p'-DDT	47	22	46.8	0.07	0.01-1.11
p,p'-DDT	47	32	68.1	0.29	0.01-3.28
DDTR	47	34	72.3	0.67	0.03-7.20
Dieldrin	47	12	25.5	0.02	0.01-0.24
Endrin	47	5	10.6	0.01	0.01-0.29
Endrin ketone	47	2	4.3	<0.01	0.10-0.13
p,p'-TDE	47	27	57.5	0.07	0.01-1.19
Toxaphene	47	8	17.0	0.27	0.32-3.40
Trifluralin	47	4	8.5	0.01	0.01-0.20
CALIFORNIA					
Aldrin	65	1	1.5	<0.01	0.03
Arsenic	65	65	100.0	5.15	0.74-23.67
Carbophenothion	17	1	5.9	0.01	0.23
Chlordane	65	2	3.1	0.01	0.10-0.32
DCPA	65	1	1.5	0.01	0.54
o,p'-DDE	65	24	36.9	0.01	0.01-0.14

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
CALIFORNIA—Continued					
p,p'-DDE	65	55	84.6	0.37	0.01-5.93
o,p'-DDT	65	32	49.2	0.08	0.01-1.33
p,p'-DDT	65	48	73.9	0.54	0.01-11.09
DDTR	65	55	84.6	1.47	0.01-41.81
Diazinon	17	1	5.9	<0.01	0.02
Dicofol	65	6	9.2	0.02	0.03-1.07
Dieldrin	65	20	30.8	0.02	0.01-0.31
Endosulfan (I)	65	1	1.5	<0.01	0.01
Endosulfan (II)	65	5	7.7	<0.01	0.01-0.09
Endosulfan sulfate	65	5	7.7	0.01	0.02-0.15
Endrin	65	9	13.9	0.01	0.01-0.16
Heptachlor epoxide	65	8	12.3	<0.01	0.01-0.03
Lindane	65	2	3.1	<0.01	0.02
Ethyl parathion	17	1	5.9	<0.01	0.02
o,p'-TDE	65	13	20.0	0.09	0.01-4.52
p,p'-TDE	65	40	61.5	0.38	0.01-20.13
Toxaphene	65	10	15.4	0.16	0.16-2.07
Trifluralin	65	7	10.8	<0.01	0.01-0.10
COLORADO					
Aldrin	60	1	1.7	<0.01	0.02
Arsenic	58	58	100.0	4.60	1.78-9.46
p,p'-DDE	60	7	11.7	0.01	0.01-0.17
o,p'-DDT	60	2	3.3	<0.01	0.01-0.03
p,p'-DDT	60	5	8.3	0.01	0.01-0.22
DDTR	60	8	13.3	0.01	0.01-0.42
Dieldrin	60	5	8.3	0.01	0.01-0.61
Endrin	60	3	5.0	<0.01	0.01-0.02
Endrin ketone	60	1	1.7	<0.01	0.05
p,p'-TDE	60	1	1.7	<0.01	0.01
CONNECTICUT					
Arsenic	2	2	100.0	3.96	2.33-5.59
p,p'-DDE	2	1	50.0	0.01	0.01
p,p'-DDT	2	1	50.0	0.03	0.05
DDTR	2	1	50.0	0.03	0.06
Dieldrin	2	1	50.0	0.01	0.01
DELAWARE					
Arsenic	3	3	100.0	2.97	0.95-5.88
p,p'-DDE	3	1	33.3	<0.01	0.01
DDTR	3	1	33.3	<0.01	0.01
Dieldrin	3	1	33.3	<0.01	0.01
FLORIDA					
Aldrin	18	1	5.6	0.03	0.47
Arsenic	18	16	88.9	0.77	0.25-3.08
Chlordane	18	9	50.0	0.36	0.04-3.32
o,p'-DDE	18	2	11.1	0.01	0.03-0.06
p,p'-DDE	18	13	72.2	0.25	0.01-2.40
o,p'-DDT	18	9	50.0	0.10	0.01-0.98
p,p'-DDT	18	14	77.8	0.37	0.01-2.08
DDTR	18	14	77.8	0.85	0.01-5.03

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
FLORIDA—Continued					
Diazinon	5	1	20.0	0.03	0.15
Dieldrin	18	7	38.9	0.08	0.01-0.52
Endrin	18	2	11.1	0.03	0.13-0.38
Endrin aldehyde	18	1	5.6	<0.01	0.03
Endrin ketone	18	1	5.6	<0.01	0.03
Ethion	5	1	20.0	0.01	0.03
Heptachlor	18	1	5.6	<0.01	0.05
Heptachlor epoxide	18	3	16.7	0.01	0.01-0.07
Ethyl parathion	5	2	40.0	0.60	0.01-3.01
o,p'-TDE	18	1	5.6	0.02	0.34
p,p'-TDE	18	11	61.1	0.11	0.01-0.64
Toxaphene	18	2	11.1	0.08	0.62-0.77
Trifluralin	18	1	5.6	<0.01	0.03
GEORGIA					
Arsenic	29	29	100.0	2.61	0.37-10.72
Chlordane	22	1	4.6	0.01	0.19
2,4-D	3	1	33.3	<0.01	0.01
o,p'-DDE	22	5	22.7	0.01	0.01-0.08
p,p'-DDE	22	20	90.9	0.18	0.01-1.04
o,p'-DDT	22	13	59.1	0.09	0.01-0.73
p,p'-DDT	22	18	81.8	0.56	0.01-4.64
DDTR	22	21	95.5	0.96	0.01-6.31
DEF	22	1	4.6	0.01	0.12
Dieldrin	22	4	18.2	<0.01	0.01-0.03
Endrin	22	1	4.6	0.02	0.42
Heptachlor epoxide	22	1	4.6	<0.01	0.02
PCNB	22	1	4.6	0.03	0.69
o,p'-TDE	22	1	4.6	0.02	0.34
p,p'-TDE	22	15	68.2	0.10	0.01-1.23
Toxaphene	22	8	36.4	0.60	0.43-5.63
Trifluralin	22	3	13.6	<0.01	0.02-0.04
IDAHO					
Arsenic	33	32	97.0	3.22	0.47-8.58
Chlordane	33	2	6.1	<0.01	0.03-0.07
p,p'-DDE	33	8	24.2	0.01	0.01-0.09
o,p'-DDT	33	6	18.2	0.01	0.01-0.13
p,p'-DDT	33	8	24.2	0.04	0.01-0.67
DDTR	33	8	24.2	0.07	0.02-1.03
Dieldrin	33	3	9.1	0.01	0.03-0.11
Heptachlor epoxide	33	1	3.0	<0.01	0.01
o,p'-TDE	33	3	9.1	<0.01	0.01
p,p'-TDE	33	6	18.2	0.01	0.01-0.15
Trifluralin	33	2	6.1	0.01	0.01-0.24
ILLINOIS					
Aldrin	142	60	42.3	0.13	0.01-2.24
Arsenic	142	142	100.0	8.05	1.54-33.40
Atrazine	43	2	4.7	<0.01	0.02-0.10
Chlordane	142	36	25.4	0.23	0.02-5.20
p,p'-DDE	142	16	11.3	<0.01	0.01-0.05
o,p'-DDT	142	4	2.8	<0.01	0.01-0.02
p,p'-DDT	142	12	8.5	<0.01	0.01-0.06
DDTR	142	16	11.3	0.01	0.01-0.29

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
ILLINOIS—Continued					
Dieldrin	142	87	61.3	0.11	0.01-1.42
Heptachlor	142	31	21.8	0.03	0.01-0.59
Heptachlor epoxide	142	36	25.4	0.02	0.01-1.08
Isodrin	142	2	1.4	<0.01	0.02
o,p'-TDE	142	1	0.7	<0.01	0.06
p,p'-TDE	142	5	3.5	<0.01	0.01-0.16
Trifluralin	142	2	1.4	<0.01	0.05-0.16
INDIANA					
Aldrin	78	13	16.7	0.07	0.01-3.06
Arsenic	78	78	100.0	7.88	1.28-19.65
Chlordane	78	4	5.1	0.02	0.07-0.53
p,p'-DDE	78	1	1.3	<0.01	0.03
o,p'-DDT	78	2	2.6	<0.01	0.01-0.03
p,p'-DDT	78	2	2.6	<0.01	0.02-0.09
DDTR	78	2	2.6	<0.01	0.06-0.14
Dieldrin	78	21	26.9	0.03	0.01-0.58
Heptachlor	78	2	2.6	<0.01	0.02-0.08
Heptachlor epoxide	78	1	1.3	<0.01	0.02
Isodrin	78	1	1.3	<0.01	0.03
p,p'-TDE	78	2	2.6	<0.01	0.01
Trifluralin	78	1	1.3	<0.01	0.03
IOWA					
Aldrin	151	48	31.8	0.04	0.01-1.37
Arsenic	152	152	100.0	7.51	0.86-107.45
Atrazine	48	13	27.1	0.05	0.01-1.55
Chlordane	151	32	21.2	0.13	0.04-6.30
p,p'-DDE	151	21	13.9	0.01	0.01-0.18
o,p'-DDT	151	6	4.0	<0.01	0.01-0.05
p,p'-DDT	151	23	15.2	0.01	0.01-0.34
DDTR	151	25	16.6	0.03	0.01-0.60
Dieldrin	151	81	53.6	0.06	0.01-0.42
Heptachlor	151	14	9.3	0.02	0.01-0.97
Heptachlor epoxide	151	31	20.5	0.01	0.01-0.33
Isodrin	151	2	1.3	<0.01	0.01-0.02
o,p'-TDE	151	1	0.7	<0.01	0.10
p,p'-TDE	151	8	5.3	<0.01	0.01-0.50
Trifluralin	151	5	3.3	<0.01	0.02-0.08
KENTUCKY					
Aldrin	31	8	25.8	0.03	0.01-0.42
Arsenic	31	31	100.0	8.41	2.60-12.80
Chlordane	31	4	12.9	0.02	0.06-0.27
o,p'-DDE	31	1	3.2	<0.01	0.03
p,p'-DDE	31	5	16.1	0.01	0.01-0.17
o,p'-DDT	31	3	9.7	0.02	0.01-0.34
p,p'-DDT	31	6	19.4	0.04	0.02-1.00
DDTR	31	6	19.4	0.08	0.03-1.84
Dieldrin	31	17	54.8	0.06	0.01-0.65
Heptachlor	31	2	6.5	<0.01	0.01
Heptachlor epoxide	31	1	3.2	<0.01	0.02
Isodrin	31	2	6.5	<0.01	0.01
o,p'-TDE	31	1	3.2	<0.01	0.02
p,p'-TDE	31	4	12.9	0.01	0.02-0.28

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
LOUISIANA					
Aldrin	27	5	18.5	<0.01	0.01-0.02
Arsenic	27	26	96.3	2.15	0.26-6.34
Chlordane	27	1	3.7	<0.01	0.11
o,p'-DDE	27	2	7.4	<0.01	0.04-0.05
p,p'-DDE	27	12	44.4	0.19	0.01-2.55
o,p'-DDT	27	9	33.3	0.10	0.02-1.45
p,p'-DDT	27	13	48.2	0.61	0.02-7.17
DDTR	27	13	48.2	0.99	0.03-10.99
Dieldrin	27	10	37.0	0.02	0.01-0.13
Endrin	27	1	3.7	<0.01	0.06
Endrin ketone	27	1	3.7	<0.01	0.02
p,p'-TDE	27	9	33.3	0.08	0.02-1.63
Toxaphene	27	4	14.8	0.57	0.59-11.72
Trifluralin	27	1	3.7	<0.01	0.07
MAINE					
Arsenic	8	8	100.0	16.01	5.06-44.06
Chlordane	8	1	12.5	0.02	0.12
p,p'-DDE	8	6	75.0	0.12	0.02-0.36
o,p'-DDT	8	5	62.5	0.13	0.02-0.46
p,p'-DDT	8	6	75.0	0.54	0.04-1.87
DDTR	8	6	75.0	0.85	0.08-2.86
Endrin	8	1	12.5	0.02	0.15
Heptachlor	8	1	12.5	<0.01	0.01
Heptachlor epoxide	8	1	12.5	<0.01	0.01
p,p'-TDE	8	6	75.0	0.06	0.01-0.19
MARYLAND					
Arsenic	12	12	100.0	5.69	3.40-11.90
Chlordane	12	1	8.3	0.01	0.09
p,p'-DDE	12	2	16.7	<0.01	0.02
p,p'-DDT	12	1	8.3	<0.01	0.03
DDTR	12	2	16.7	0.01	0.02-0.05
Dieldrin	12	1	7.3	0.01	0.06
Heptachlor epoxide	12	1	8.3	<0.01	0.02
MASSACHUSETTS					
Arsenic	2	2	100.0	9.75	7.35-12.15
p,p'-DDE	2	1	50.0	0.17	0.34
o,p'-DDT	2	1	50.0	0.10	0.20
p,p'-DDT	2	1	50.0	0.49	0.97
DDTR	2	1	50.0	0.78	1.55
p,p'-TDE	2	1	50.0	0.02	0.04
MICHIGAN					
Aldrin	51	2	3.9	<0.01	0.01-0.10
Arsenic	52	52	100.0	4.85	0.13-11.94
Atrazine	13	1	7.7	0.01	0.18
o,p'-DDE	51	4	7.8	<0.01	0.01-0.14
p,p'-DDE	51	12	23.5	0.16	0.01-4.58
o,p'-DDT	51	3	5.9	0.18	0.15-6.29
p,p'-DDT	51	5	9.8	1.10	0.01-35.92
DDTR	51	12	23.5	2.09	0.01-78.36

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
MICHIGAN—Continued					
Dieldrin	51	11	21.6	0.05	0.01-1.01
Endosulfan (I)	51	2	3.9	0.01	0.03-0.24
Endosulfan sulfate	51	2	3.9	0.02	0.25-0.94
Endrin	51	1	2.0	<0.01	0.01
p,p'-TDE	51	5	9.8	0.65	0.02-31.43
MISSISSIPPI					
Arsenic	30	30	100.0	5.70	1.10-16.90
o,p'-DDE	29	9	31.0	0.01	0.01-0.08
p,p'-DDE	29	26	89.7	0.31	0.01-1.43
o,p'-DDT	29	22	75.9	0.20	0.02-1.35
p,p'-DDT	29	26	89.7	1.36	0.01-9.28
DDTR	29	26	89.7	2.06	0.03-13.14
Dieldrin	29	10	34.5	0.01	0.02-0.10
Endrin	29	1	3.5	0.01	0.19
Endrin ketone	29	1	3.5	<0.01	0.11
Lindane	29	2	6.9	<0.01	0.01-0.04
o,p'-TDE	29	2	6.9	0.03	0.33-0.49
p,p'-TDE	29	20	69.0	0.15	0.01-0.81
Toxaphene	29	14	48.3	0.78	0.10-8.80
Trifluralin	29	6	20.7	0.02	0.02-0.25
MISSOURI					
Aldrin	82	18	22.0	0.05	0.01-1.59
Arsenic	81	80	98.8	5.99	0.49-24.51
Chlordane	82	6	7.3	0.03	0.17-0.60
p,p'-DDE	82	3	3.7	<0.01	0.01
o,p'-DDT	82	2	2.4	<0.01	0.01-0.02
p,p'-DDT	82	3	3.7	<0.01	0.02-0.09
DDTR	82	3	3.7	<0.01	0.03-0.12
Dieldrin	82	26	31.7	0.04	0.01-0.55
Endrin	82	1	1.2	<0.01	0.01
Heptachlor	82	5	6.1	<0.01	0.01-0.04
Heptachlor epoxide	82	5	6.1	<0.01	0.01-0.06
Isodrin	82	1	1.2	<0.01	0.03
Toxaphene	82	1	1.2	0.04	3.15
Trifluralin	82	5	6.1	<0.01	0.02-0.10
NEBRASKA					
Aldrin	106	2	1.9	<0.01	0.01
Arsenic	106	106	100.0	5.81	0.33-15.80
Atrazine	72	12	16.7	<0.01	0.01-0.12
Chlordane	106	11	10.4	0.01	0.03-0.18
p,p'-DDE	106	14	13.2	0.01	0.01-0.10
o,p'-DDT	106	6	5.7	<0.01	0.01-0.08
p,p'-DDT	106	12	11.3	0.01	0.01-0.19
DDTR	106	16	15.1	0.01	0.02-0.31
Dicofol	106	2	1.9	<0.01	0.10
Dieldrin	106	37	34.9	0.02	0.01-0.19
Endrin	106	1	0.9	<0.01	0.02
Heptachlor	106	1	0.9	<0.01	0.01
Heptachlor epoxide	106	12	11.3	<0.01	0.01-0.03
Malathion	2	1	50.0	0.18	0.36
p,p'-TDE	106	4	3.8	<0.01	0.01-0.05

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
NEVADA					
Arsenic	2	2	100.0	2.32	1.77-2.86
NEW HAMPSHIRE					
Arsenic	2	2	100.0	5.35	1.31-9.38
p,p'-DDE	2	1	50.0	0.02	0.03
DDTR	2	1	50.0	0.02	0.03
NEW JERSEY					
Arsenic	5	5	100.0	11.72	4.55-17.21
o,p'-DDE	5	1	20.0	<0.01	0.02
p,p'-DDE	5	2	40.0	0.17	0.18-0.66
o,p'-DDT	5	1	20.0	0.06	0.28
p,p'-DDT	5	2	40.0	0.24	0.05-1.17
DDTR	5	2	40.0	0.55	0.26-2.48
Dieldrin	5	2	40.0	0.05	0.05-0.21
Endosulfan (II)	5	1	20.0	<0.01	0.02
Endosulfan sulfate	5	1	20.0	0.02	0.11
Heptachlor epoxide	5	1	20.0	<0.01	0.01
Lindane	5	1	20.0	0.01	0.03
Ethyl parathion	1	1	100.0	0.02	0.02
o,p'-TDE	5	1	20.0	0.02	0.09
p,p'-TDE	5	2	40.0	0.06	0.03-0.26
NEW MEXICO					
Arsenic	10	10	100.0	4.64	0.66-15.82
p,p'-DDE	10	4	40.0	0.02	0.01-0.11
o,p'-DDT	10	1	10.0	<0.01	0.01
p,p'-DDT	10	4	40.0	0.01	0.01-0.03
DDTR	10	4	40.0	0.02	0.02-0.15
Dieldrin	10	1	10.0	<0.01	0.01
NEW YORK					
Arsenic	37	35	94.6	9.38	1.24-43.90
Chlordane	38	1	2.6	0.08	3.19
o,p'-DDE	38	3	7.9	<0.01	0.01-0.06
p,p'-DDE	38	15	39.5	0.23	0.01-3.70
o,p'-DDT	38	11	29.0	0.07	0.01-1.45
p,p'-DDT	38	13	34.2	0.53	0.02-7.67
DDTR	38	15	39.5	0.91	0.01-13.29
Dieldrin	38	13	34.2	0.05	0.01-0.96
Endrin	38	1	2.6	0.01	0.56
Endrin ketone	38	1	2.6	<0.01	0.05
Lindane	38	3	7.9	0.01	0.01-0.23
Methoxychlor	38	1	2.6	0.01	0.28
o,p'-TDE	38	2	5.3	0.01	0.06-0.37
p,p'-TDE	38	10	26.3	0.07	0.01-1.49
NORTH CAROLINA					
Aldrin	31	3	9.7	0.05	0.01-1.12
Arsenic	27	27	100.0	6.18	0.73-22.00
Chlordane	31	1	3.2	<0.01	0.11

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
NORTH CAROLINA—Continued					
o,p'-DDE	31	6	19.4	<0.01	0.01-0.03
p,p'-DDE	31	22	71.0	0.08	0.01-0.44
o,p'-DDT	31	14	45.2	0.07	0.03-0.83
p,p'-DDT	31	19	61.3	0.28	0.01-1.75
DDTR	31	22	71.0	0.53	0.02-2.88
Dieldrin	31	10	32.3	0.08	0.01-1.53
Endrin	31	2	6.5	<0.01	0.01-0.08
Heptachlor	31	2	6.5	<0.01	0.01-0.02
Heptachlor epoxide	31	4	12.9	<0.01	0.01-0.03
Isodrin	31	1	3.2	<0.01	0.01
Ethyl parathion	6	1	16.7	<0.01	0.02
o,p'-TDE	31	11	35.5	0.03	0.03-0.17
p,p'-TDE	31	19	61.3	0.07	0.01-0.27
Toxaphene	31	7	22.6	0.28	0.34-3.20
Trifluralin	31	2	6.5	<0.01	0.03-0.11
NORTH DAKOTA					
Aldrin	157	1	0.6	<0.01	0.03
Arsenic	158	158	100.0	8.50	0.98-37.53
Chlordane	157	3	1.9	<0.01	0.08-0.15
p,p'-DDE	157	10	6.4	<0.01	0.01-0.14
o,p'-DDT	157	5	3.2	<0.01	0.01-0.19
p,p'-DDT	157	8	5.1	0.01	0.01-0.56
DDTR	157	10	6.4	0.01	0.01-0.95
Dieldrin	157	12	7.6	<0.01	0.01-0.20
Endrin	157	1	0.6	<0.01	0.01
Heptachlor epoxide	157	3	1.9	<0.01	0.02-0.07
p,p'-TDE	157	7	4.5	<0.01	0.01-0.06
OHIO					
Aldrin	68	10	14.7	0.03	0.01-0.74
Arsenic	69	69	100.0	11.23	1.15-41.49
Chlordane	68	3	4.4	0.01	0.01-0.71
o,p'-DDE	68	1	1.5	<0.01	0.20
p,p'-DDE	68	11	16.2	0.03	0.01-1.77
o,p'-DDT	68	2	2.9	0.01	0.19-0.22
p,p'-DDT	68	6	8.8	0.04	0.01-1.27
DDTR	68	11	16.2	0.08	0.01-3.38
Dieldrin	68	19	27.9	0.02	0.01-0.30
Endosulfan (I)	68	1	1.5	<0.01	0.07
Endosulfan (II)	68	1	1.5	<0.01	0.29
Endosulfan sulfate	68	1	1.5	0.01	0.40
Heptachlor	68	2	2.9	<0.01	0.01
Heptachlor epoxide	68	1	1.5	<0.01	0.01
Isodrin	68	2	2.9	<0.01	0.01-0.03
Lindane	68	1	1.5	0.01	0.35
p,p'-TDE	68	3	4.4	<0.01	0.04-0.12
Trifluralin	68	1	1.5	<0.01	0.06
OKLAHOMA					
Arsenic	62	60	96.8	3.30	0.24-14.58
Chlordane	64	1	1.6	<0.01	0.07
p,p'-DDE	64	10	15.6	<0.01	0.01-0.09
o,p'-DDT	64	1	1.6	<0.01	0.01
p,p'-DDT	64	9	14.1	<0.01	0.01-0.09
DDTR	64	10	15.6	0.01	0.02-0.17

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
OKLAHOMA—Continued					
Dieldrin	64	2	3.1	<0.01	0.01
Heptachlor epoxide	64	1	1.6	<0.01	0.01
p,p'-TDE	64	2	3.1	<0.01	0.01-0.02
Trifluralin	64	1	1.6	<0.01	0.03
PENNSYLVANIA					
Arsenic	29	29	100.0	10.80	2.96-64.94
Chlordane	29	6	20.7	0.07	0.02-0.92
o,p'-DDE	29	1	3.5	<0.01	0.08
p,p'-DDE	29	9	31.0	0.07	0.01-1.52
o,p'-DDT	29	5	17.2	0.03	0.01-0.67
p,p'-DDT	29	8	27.6	0.12	0.01-2.99
DDTR	29	11	37.9	0.27	0.01-5.50
Dicofol	29	1	3.5	0.02	0.53
Dieldrin	29	10	34.5	0.02	0.01-0.14
Endosulfan (II)	29	1	3.5	<0.01	0.02
Endosulfan sulfate	29	1	3.5	<0.01	0.01
Heptachlor epoxide	29	4	13.8	<0.01	0.01-0.03
Ethyl parathion	3	1	33.3	<0.01	0.01
o,p'-TDE	29	4	13.8	0.01	0.03-0.20
p,p'-TDE	29	7	24.1	0.04	0.01-0.55
Trifluralin	29	2	6.9	<0.01	0.01-0.07
RHODE ISLAND					
Arsenic	1	1	100.0	21.30	21.30
p,p'-DDE	1	1	100.0	0.23	0.23
o,p'-DDT	1	1	100.0	0.25	0.25
p,p'-DDT	1	1	100.0	2.46	2.46
DDTR	1	1	100.0	3.00	3.00
Dieldrin	1	1	100.0	0.11	0.11
p,p'-TDE	1	1	100.0	0.06	0.06
SOUTH CAROLINA					
Aldrin	17	1	5.9	0.01	0.14
Arsenic	17	17	100.0	3.28	0.53-19.54
2,4-D	2	1	50.0	0.02	0.03
o,p'-DDE	17	7	41.2	0.01	0.01-0.05
p,p'-DDE	17	14	82.4	0.24	0.01-0.93
o,p'-DDT	17	12	70.6	0.15	0.01-0.95
p,p'-DDT	17	11	64.7	0.64	0.12-3.15
DDTR	17	15	88.2	1.17	0.01-4.78
Dieldrin	17	3	17.7	0.04	0.02-0.56
Endrin	17	3	17.7	<0.01	0.01-0.05
Heptachlor epoxide	17	3	17.7	<0.01	0.01
Lindane	17	1	5.9	<0.01	0.01
o,p'-TDE	17	5	29.4	0.03	0.03-0.19
p,p'-TDE	17	14	82.4	0.10	0.01-0.34
Toxaphene	17	1	5.9	0.10	1.74
Trifluralin	17	5	29.4	0.01	0.01-0.08
SOUTH DAKOTA					
Arsenic	101	101	100.0	5.80	0.47-34.54
Chlordane	106	3	2.8	0.01	0.10-0.66
p,p'-DDE	106	2	1.9	<0.01	0.01-0.03

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
SOUTH DAKOTA—Continued					
o,p'-DDT	106	2	1.9	<0.01	0.01-0.03
p,p'-DDT	106	2	1.9	<0.01	0.02-0.04
DDTR	106	4	3.8	<0.01	0.01-0.10
Dieldrin	106	9	8.5	0.01	0.01-0.25
Heptachlor	106	1	0.9	<0.01	0.01
Heptachlor epoxide	106	3	2.8	<0.01	0.01-0.03
Lindane	106	3	2.8	<0.01	0.01-0.02
p,p'-TDE	106	1	0.9	<0.01	0.02
TENNESSEE					
Arsenic	27	27	100.0	8.05	2.31-15.63
Chlordane	27	1	3.7	0.01	0.20
p,p'-DDE	27	10	37.0	0.02	0.01-0.26
o,p'-DDT	27	7	25.9	0.01	0.01-0.08
p,p'-DDT	27	10	37.0	0.05	0.01-0.38
DDTR	27	11	40.7	0.11	0.01-0.70
Dieldrin	27	6	22.2	<0.01	0.01-0.03
Endrin	27	1	3.7	<0.01	0.02
p,p'-TDE	27	6	22.2	0.03	0.02-0.36
Toxaphene	27	4	14.8	0.14	0.13-2.19
Trifluralin	27	2	7.4	<0.01	0.04-0.05
UTAH					
Arsenic	12	11	91.7	4.16	0.62-12.66
Chlordane	12	4	33.3	0.04	0.02-0.25
p,p'-DDE	12	2	16.7	<0.01	0.01-0.02
p,p'-DDT	12	1	8.3	<0.01	0.03
DDTR	12	2	16.7	0.01	0.01-0.05
Dieldrin	12	2	16.7	0.01	0.02-0.15
Heptachlor	12	2	16.7	0.02	0.02-0.26
Heptachlor epoxide	12	3	25.0	0.01	0.02-0.05
VERMONT					
Arsenic	4	4	100.0	1.79	0.99-2.30
p,p'-DDE	5	1	20.0	<0.01	0.01
DDTR	5	1	20.0	<0.01	0.01
Dieldrin	5	1	20.0	<0.01	0.01
VIRGINIA					
Aldrin	21	1	4.8	<0.01	0.01
Arsenic	20	20	100.0	3.34	0.69-12.34
Chlordane	21	5	23.8	0.01	0.01-0.11
p,p'-DDE	21	11	52.4	0.02	0.01-0.22
o,p'-DDT	21	4	19.1	0.01	0.01-0.17
p,p'-DDT	21	8	38.1	0.11	0.01-1.31
DDTR	21	11	52.4	0.17	0.01-1.75
Dieldrin	21	6	28.6	0.08	0.01-1.60
Heptachlor epoxide	21	4	19.1	0.01	0.01-0.05
Malathion	1	1	100.0	0.04	0.04
Ethyl parathion	1	1	100.0	0.90	0.90
o,p'-TDE	21	1	4.8	<0.01	0.07
p,p'-TDE	21	7	33.3	0.02	0.01-0.19
Toxaphene	21	1	4.7	0.01	0.28

TABLE 3.—*Pesticide residues in cropland soil from 43 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
WASHINGTON					
Aldrin	45	2	4.4	<0.01	0.09-0.10
Arsenic	45	45	100.0	2.61	0.71-7.02
2,4-D	6	1	16.7	<0.01	0.01
o,p'-DDE	45	2	4.4	<0.01	0.01-0.09
p,p'-DDE	45	10	22.2	0.17	0.01-6.99
o,p'-DDT	45	6	13.3	0.06	0.01-2.58
p,p'-DDT	45	10	22.2	0.46	0.01-19.75
DDTR	45	11	24.4	0.72	0.01-30.69
Dieldrin	45	8	17.8	0.02	0.01-0.30
o,p'-TDE	45	1	2.2	<0.01	0.17
p,p'-TDE	45	3	6.7	0.03	0.01-1.11
Toxaphene	45	1	2.2	0.02	0.73
Trifluralin	45	1	2.2	<0.01	0.08
WEST VIRGINIA					
Arsenic	6	6	100.0	6.33	4.36-8.17
Chlordane	6	3	50.0	0.21	0.09-0.78
p,p'-DDE	6	2	33.3	0.02	0.04-0.10
p,p'-DDT	6	2	33.3	0.01	0.01-0.07
DDTR	6	2	33.3	0.04	0.05-0.17
Dieldrin	6	1	16.7	0.04	0.23
Heptachlor epoxide	6	3	50.0	0.06	0.08-0.18
WISCONSIN					
Aldrin	67	5	7.5	<0.01	0.01-0.04
Arsenic	68	68	100.0	3.78	0.34-10.01
Chlordane	67	3	4.5	0.01	0.04-0.32
o,p'-DDE	67	1	1.5	<0.01	0.02
p,p'-DDE	67	9	13.4	0.01	0.01-0.27
o,p'-DDT	67	3	4.5	0.01	0.05-0.20
p,p'-DDT	67	7	10.5	0.01	0.01-0.30
DDTR	67	9	13.4	0.02	0.01-0.71
Dieldrin	67	9	13.4	0.01	0.01-0.17
Heptachlor	67	2	3.0	<0.01	0.01
Heptachlor epoxide	67	2	3.0	<0.01	0.01
p,p'-TDE	67	4	6.0	<0.01	0.01-0.12
Trifluralin	67	1	1.5	<0.01	0.01
WYOMING					
Arsenic	17	14	82.4	1.71	0.40-10.88
Chlordane	17	4	23.5	0.05	0.03-0.48
Dieldrin	17	6	35.3	0.02	0.02-0.19
Heptachlor epoxide	17	3	17.7	0.01	0.02-0.05

[1] One sample per site.
[2] Percent based on number of sites with residues greater than or equal to the sensitivity limits.

TABLE 4.—*Pesticide residues in noncropland soil from 11 States—FY 1969*

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
ARIZONA					
Arsenic	44	44	100.0	6.63	1.35-30.64
Chlordane	44	1	2.3	<0.01	0.08
p,p'-DDE	44	8	18.2	<0.01	0.01-0.06
p,p'-DDT	44	1	2.3	<0.01	0.03
DDTR	44	8	18.2	<0.01	0.01-0.09
Dieldrin	44	1	2.3	<0.01	0.03
GEORGIA					
Arsenic	19	18	94.7	1.47	0.53-4.29
p,p'-DDE	10	6	60.0	0.02	0.01-0.07
o,p'-DDT	10	2	20.0	<0.01	0.01-0.02
p,p'-DDT	10	5	50.0	0.02	0.01-0.10
DDTR	10	7	70.0	0.05	0.01-0.12
Dieldrin	10	1	10.0	<0.01	0.01
p,p'-TDE	10	1	10.0	<0.01	0.01
IDAHO					
Arsenic	26	26	100.0	7.73	1.01-39.07
p,p'-DDE	26	3	11.5	<0.01	0.01
o,p'-DDT	26	1	3.9	<0.01	0.02
p,p'-DDT	26	1	3.9	<0.01	0.06
DDTR	26	3	11.5	0.01	0.01-0.11
p,p'-TDE	26	1	3.9	<0.01	0.02
IOWA					
Aldrin	7	1	14.3	<0.01	0.02
Arsenic	7	7	100.0	7.08	1.71-17.39
MAINE					
Arsenic	8	8	100.0	5.14	1.40-13.00
p,p'-DDE	11	1	9.1	0.02	0.18
o,p'-DDT	11	1	9.1	<0.01	0.03
p,p'-DDT	11	1	9.1	0.02	0.23
DDTR	11	1	9.1	0.06	0.62
p,p'-TDE	11	1	9.1	0.02	0.18
MARYLAND					
Arsenic	3	3	100.0	8.43	5.20-11.97
p,p'-DDE	3	1	33.3	0.02	0.05
o,p'-DDT	3	1	33.3	0.01	0.03
p,p'-DDT	3	2	66.7	0.05	0.03-0.11
DDTR	3	2	66.7	0.09	0.03-0.23
p,p'-TDE	3	1	33.3	0.01	0.04
NEBRASKA					
Arsenic	17	16	94.1	2.18	0.33-8.42
Chlordane	19	1	5.3	<0.01	0.04
p,p'-DDE	19	3	15.8	<0.01	0.01-0.04
o,p'-DDT	19	1	5.3	<0.01	0.01
p,p'-DDT	19	1	5.3	<0.01	0.02

TABLE 4.—*Pesticide residues in noncropland soil from 11 States—FY 1969*—Continued

Compound	Number of Samples Analyzed [1]	Number of Positive Samples	Percent Positive Sites [2]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
NEBRASKA—Continued					
DDTR	19	3	15.8	<0.01	0.01-0.07
Dicofol	19	2	10.5	0.02	0.10-0.29
Dieldrin	19	2	10.5	<0.01	0.01
Heptachlor epoxide	19	1	5.3	<0.01	0.01
VIRGINIA					
Arsenic	10	10	100.0	4.07	0.50-12.42
p,p'-DDT	13	3	23.1	0.01	0.03-0.07
DDTR	13	3	23.1	0.01	0.03-0.09
Dieldrin	13	2	15.4	0.01	0.03-0.09
p,p'-TDE	13	1	7.7	<0.01	0.02
WASHINGTON					
Arsenic	21	21	100.0	6.94	1.58-54.17
p,p'-DDE	21	3	14.3	<0.01	0.01-0.02
p,p'-DDT	21	2	9.5	<0.01	0.01
DDTR	21	3	14.3	<0.01	0.01-0.03
WEST VIRGINIA					
Arsenic	6	6	100.0	5.16	2.67-13.26
p,p'-DDE	8	1	12.5	<0.01	0.02
p,p'-DDT	8	1	12.5	0.01	0.05
DDTR	8	1	12.5	0.01	0.08
Dieldrin	8	1	12.5	0.01	0.04
p,p'-TDE	8	1	12.5	<0.01	0.01
WYOMING					
Arsenic	37	36	97.3	2.73	0.35-19.33
Chlordane	37	1	2.7	0.01	0.50
o,p'-DDE	37	1	2.7	<0.01	0.02
p,p'-DDE	37	1	2.7	0.01	0.31
o,p'-DDT	37	1	2.7	<0.01	0.05
p,p'-DDT	37	1	2.7	<0.01	0.18
DDTR	37	1	2.7	0.02	0.56
Dieldrin	37	1	2.7	<0.01	0.02
Heptachlor epoxide	37	1	2.7	<0.01	0.01
Toxaphene	37	1	2.7	0.01	0.52

[1] One sample per site.

[2] Percent based on number of sites with residues greater than or equal to the sensitivity limits.

TABLE 5.—*Arsenic residue data for the 12 States having the highest residue levels—FY 1969*

State	Number of Samples Analyzed	Percent Positive Sites [1]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
Arkansas	47	100.0	9.0	1.7-28.2
Kentucky	31	100.0	8.4	2.6-12.8
New England [2]	19	100.0	10.2	1.0-14.1
New York	37	94.6	9.4	1.2-43.9
North Dakota	158	100.0	8.5	1.0-37.5
Ohio	69	100.0	11.2	1.2-41.5
Pennsylvania	29	100.0	10.8	3.0-64.9

[1] Percent based on number of sites with residues greater than or equal to the sensitivity limits.
[2] Connecticut, Maine, Massachusetts, New Hampshire, Rhode Island, and Vermont.

TABLE 6.—*Pesticide residue data for 5 States having the highest DDTR residue levels—FY 1969*

State	Number of Samples Analyzed	Percent Positive Sites [1]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
Alabama	22	90.9	1.13	0.05-8.08
California	65	84.6	1.47	0.01-41.81
Michigan	51	23.5	2.09	0.01-78.36
Mississippi	29	89.7	2.06	0.03-13.14
South Carolina	17	88.2	1.17	0.01-4.78

[1] Percent based on number of sites with residues greater than or equal to the sensitivity limits.

TABLE 7.—*Residue data for the 7 States with the highest dieldrin residue levels—FY 1969*

State	Number of Samples Analyzed	Percent Positive Sites [1]	Mean Residue Level (ppm)	Range of Detected Residues (ppm)
Florida	18	38.9	0.08	0.01-0.52
Illinois	142	61.3	0.11	0.01-1.42
Iowa	151	53.6	0.06	0.01-0.42
Kentucky	31	54.8	0.06	0.01-0.65
North Carolina	31	32.3	0.08	0.01-1.53
Virginia/West Virginia	27	25.9	0.07	0.01-1.60

[1] Percent based on number of sites with residues greater than or equal to the sensitivity limits.

TABLE 8.—*Summary of pesticides used in FY 1969 on cropland for all 43 States*

ALL STATES—1,684 SITES

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)	Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
Aldrin	4.16	1.25	0.0522	Dithane M-45	0.30	5.82	0.0173
Amiben	2.14	1.07	0.0229	Diuron	1.13	0.93	0.0105
Aramite	0.12	2.35	0.0028	DSMA	0.36	1.52	0.0054
Atrazine	7.66	1.88	0.1442	Endosulfan (I)	0.48	1.11	0.0053
Azinphosmethyl	0.59	1.70	0.0101	Endrin	0.48	2.21	0.0105
Azodrin	0.42	2.07	0.0086	EPN	0.06	1.50	0.0009
Bacillus thuringiensis	0.12	9.50	0.0113	EPTC	0.36	2.65	0.0094
Barban	0.12	0.17	0.0002	Ethion	0.24	2.06	0.0049
Benefin	0.18	1.36	0.0024	Ethylene dibromide	0.12	14.62	0.0174
Benzene hexachloride	0.06	3.00	0.0018	Falone	0.06	2.00	0.0012
Bidrin	0.24	0.18	0.0004	Ferbam	0.06	9.12	0.0054
Binapacryl	0.06	2.12	0.0013	Folex	0.06	1.50	0.0009
Bordeaux mixtures	0.06	0.50	0.0003	Heptachlor	1.96	0.33	0.0065
Cacodylic acid	0.06	0.01	0.0000	Herbisan	0.06	10.00	0.0060
Captan	11.16	0.12	0.0133	Hexachlorobenzene	0.06	0.01	0.0000
Carbaryl	1.72	3.64	0.0627	Lead arsenate	0.06	3.80	0.0023
Carbophenothion	0.18	1.83	0.0033	Lindane	0.65	0.03	0.0002
CDAA	0.89	1.78	0.0158	Linuron	0.77	0.73	0.0056
Ceresan L	1.25	0.01	0.0002	Malathion	7.54	0.17	0.0127
Ceresan M	1.48	0.01	0.0001	Maleic hydrazide	0.36	1.43	0.0051
Ceresan red	1.84	0.01	0.0003	Maneb	0.30	2.14	0.0064
Chevron RE-5353	0.30	1.72	0.0051	MCPA	1.07	0.33	0.0035
Chlordane	0.12	3.10	0.0037	Methoxychlor	2.20	0.04	0.0008
Chlorobenzilate	0.12	1.31	0.0016	Methyl demeton	0.06	1.50	0.0009
Chloroneb	0.36	0.05	0.0002	Methylmercury dicyandiamide	5.46	0.01	0.0006
Chloroxuron	0.30	1.65	0.0049	Methylmercury nitrile	0.06	0.01	0.0000
Chromophon	0.06	0.15	0.0001	Mevinphos	0.36	1.48	0.0053
CIPC	0.12	1.50	0.0018	Mirex	0.24	0.01	0.0000
Copper carbonate	0.06	0.60	0.0004	Monuron	0.06	1.60	0.0010
Copper oxide	0.18	4.23	0.0075	MSMA	0.48	1.21	0.0058
Copper oxychloride sulfate	0.12	4.68	0.0056	Nabam	0.24	1.78	0.0042
Copper-8-quinolinolate	0.06	0.01	0.0000	Naled	0.30	1.62	0.0048
Copper sulfate	0.36	13.53	0.0482	Nitralin	0.36	0.76	0.0027
Cotoran	0.48	0.74	0.0035	Nitrate	1.13	64.58	0.7286
2,4-D	15.14	0.54	0.0825	Norea	0.12	0.46	0.0006
2,4-DB	0.89	0.48	0.0042	NPA	0.36	1.01	0.0036
Dalapon	0.42	2.12	0.0088	Oxydemetonmethyl	0.18	0.40	0.0007
DDT technical	3.44	5.56	0.1915	Ethyl parathion	1.84	1.48	0.0272
DEF	0.59	1.66	0.0099	Methyl parathion	3.03	3.07	0.0929
Demeton	0.18	0.59	0.0011	PCNB	0.42	1.59	0.0066
Diazinon	1.96	1.22	0.0240	PCP	0.06	1.50	0.0009
Dicamba	0.30	0.39	0.0012	Phenylmercury urea	0.06	0.01	0.0000
Dichlone	0.12	2.00	0.0024	Phorate	0.65	2.17	0.0142
Dichloropropane	0.06	54.43	0.0323	Phosphamidon	0.12	0.13	0.0002
Dichloropropene	0.36	70.07	0.2496	Picloram	0.12	0.63	0.0007
Dichlorprop	0.06	2.00	0.0012	PMA	0.18	0.06	0.0001
Dicofol	0.42	2.12	0.0088	Polyram	0.06	10.40	0.0062
Dieldrin	1.19	0.17	0.0021	Prometryne	0.06	2.00	0.0012
Difolatan	0.06	0.01	0.0000	Propanil	0.42	3.96	0.0165
Dimetan	0.06	0.01	0.0000	Propazine	0.06	2.00	0.0012
Dimethoate	0.12	0.75	0.0009	Ramrod	1.37	1.45	0.0198
Dinitrobutylphenol	0.95	3.78	0.0359	Ro-Neet	0.12	1.88	0.0022
Dinitrocresol	0.06	3.00	0.0018	Roundup	0.12	0.78	0.0009
Dinocap	0.12	0.22	0.0003	Randox T	0.12	0.90	0.0011
Dioxathion	0.12	2.60	0.0031	Silvex	0.12	0.63	0.0007
Diphenamid	0.24	2.19	0.0052	Simazine	0.12	2.07	0.0025
Diquat	0.06	0.83	0.0005	Simetryne	0.06	2.00	0.0012
Disulfoton	1.72	1.77	0.0305	Sodium arsenite	0.24	5.25	0.0125
				Sodium chlorate	0.06	6.00	0.0036

TABLE 8.—*Summary of pesticides used in FY 1969 on cropland for all 43 States*—Continued

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
Strobane	0.12	16.50	0.0196
Sulfur	0.71	34.00	0.2423
2,4,5-T	0.18	0.83	0.0015
TCA	0.06	2.00	0.0012
TDE technical	0.36	2.31	0.0082
Tetradifon	0.12	0.50	0.0006
Thiram	1.07	0.03	0.0003
Toxaphene	1.90	9.87	0.1876
Trichlorofon	0.06	0.80	0.0005
Trifluralin	4.33	0.76	0.0327
Vernolate	0.53	1.29	0.0069
Zineb	0.18	4.90	0.0087
Ziram	0.06	0.80	0.0005

TABLE 9.—*Summary of pesticides used in FY 1969 on noncropland for all 11 States*

ALL STATES—195 SITES

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
2,4-D	0.51	2.00	0.0103
Malathion	0.51	0.61	0.0031
Mirex	0.51	0.01	0.0001

TABLE 10.—*Summary of pesticides used in FY 1969 on cropland by State*

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
ALABAMA—23 SITES			
Azodrin	4.35	0.84	0.0365
Benzene hexachloride	4.35	3.00	0.1304
Captan	21.74	0.04	0.0083
Carbaryl	4.35	0.40	0.0174
Ceresan M	4.35	0.01	0.0004
Copper sulfate	8.70	36.08	3.1374
Cotoran	4.35	1.50	0.0652
DDT technical	39.13	10.73	4.2000
DEF	4.35	1.50	0.0652
Disulfoton	8.70	0.35	0.0304
Diuron	8.70	0.95	0.0826
DSMA	4.35	1.00	0.0435
Endrin	8.70	1.20	0.1043
EPN	4.35	1.50	0.0652

TABLE 10.—*Summary of pesticides used in FY 1969 on cropland by State*—Continued

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
ALABAMA—23 SITES—Continued			
Malathion	8.70	2.50	0.2174
Ethyl parathion	4.35	1.00	0.0435
Methyl parathion	52.17	3.42	1.7848
MSMA	8.70	1.50	0.1304
Phorate	4.35	1.00	0.0435
Prometryne	4.35	2.00	0.0870
Thiram	4.35	0.02	0.0009
Toxaphene	17.39	3.45	0.6000
Trifluralin	47.83	0.61	0.2913
Vernolate	8.70	1.05	0.0913
ARIZONA—9 SITES			
Azodrin	11.11	6.25	0.6944
Captan	11.11	0.01	0.0011
Ceresan L	11.11	0.01	0.0011
Demeton	11.11	0.13	0.0144
Dieldrin	11.11	0.01	0.0011
Diuron	11.11	1.00	0.1111
Endosulfan (I)	11.11	2.00	0.2222
Naled	11.11	0.50	0.0556
Ethyl parathion	22.22	5.50	1.2222
Methyl parathion	44.44	2.75	1.2244
PCNB	11.11	0.75	0.0833
Phorate	11.11	1.50	0.1667
Strobane	11.11	15.00	1.6667
Toxaphene	11.11	2.00	0.2222
Trifluralin	22.22	1.12	0.2500
ARKANSAS—45 SITES			
Aldrin	2.22	0.25	0.0056
Captan	13.33	0.03	0.0036
Ceresan M	2.22	0.01	0.0002
Chloroxuron	6.67	1.00	0.0667
2,4-D	2.22	0.05	0.0011
2,4-DB	2.22	1.75	0.0389
DEF	2.22	1.00	0.0222
Dinitrobutylphenol	6.67	1.58	0.1056
Disulfoton	2.22	0.01	0.0002
Diuron	2.22	0.75	0.0167
DSMA	4.44	3.00	0.1333
Endrin	2.22	12.00	0.2667
Linuron	8.89	0.94	0.0833
NPA	6.67	0.54	0.0362
Nitralin	2.22	0.44	0.0098
Methyl parathion	2.22	12.00	0.2667
Propanil	4.44	5.50	0.2444
2,4,5-T	4.44	0.88	0.0389
Thiram	4.44	0.03	0.0016
Trifluralin	15.56	0.79	0.1222
CALIFORNIA—66 SITES			
Aramite	3.03	2.35	0.0712
Atrazine	1.52	2.50	0.0379

TABLE 10.—*Summary of pesticides used in FY 1969 on cropland by State*—Continued

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
CALIFORNIA—66 SITES—Continued			
Azinphosmethyl	3.03	0.48	0.0145
Bacillus thuringiensis	3.03	9.50	0.2879
Benefin	1.52	1.83	0.0277
Binapacryl	1.52	2.12	0.0321
Bordeaux mixtures	1.52	0.50	0.0076
Captan	1.52	2.30	0.0348
Carbaryl	6.06	10.76	0.6521
Carbophenothion	3.03	1.75	0.0530
Ceresan red	3.03	0.01	0.0003
Chlordane	1.52	5.00	0.0758
2,4-D	3.03	0.63	0.0189
DDT technical	13.64	2.82	0.3844
Diazinon	6.06	0.99	0.0603
Dichloropropene	4.55	8.67	0.3939
Dichlorprop	1.52	2.00	0.0303
Dicofol	7.58	2.59	0.1962
Dimethoate	1.52	1.00	0.0152
Dioxathion	3.03	2.60	0.0788
Diphenamid	1.52	2.62	0.0397
Disulfoton	3.03	4.00	0.1212
Diuron	1.52	0.75	0.0114
Dithane M-45	3.03	5.00	0.1515
Endosulfan (I)	7.58	0.99	0.0750
Ethion	3.03	1.38	0.0417
Malathion	4.55	1.65	0.0750
MCPA	3.03	0.76	0.0230
Mevinphos	9.09	1.48	0.1345
Nabam	1.52	3.50	0.0530
Naled	6.06	1.90	0.1152
Ethyl parathion	6.06	2.03	0.1230
Methyl parathion	4.55	6.10	0.2773
Propanil	3.03	3.63	0.1098
Simazine	1.52	3.75	0.0568
Simetryne	1.52	2.00	0.0303
Sulfur	7.58	35.79	2.7115
Tetradifon	3.03	0.50	0.0152
Toxaphene	6.06	9.75	0.5908
Trichlorofon	1.52	0.80	0.0121
Trifluralin	6.06	0.88	0.0530
COLORADO—60 SITES			
Aldrin	3.33	0.08	0.0027
Carbaryl	1.67	1.00	0.0167
Ceresan M	1.67	0.01	0.0002
2,4-D	10.00	0.51	0.0508
2,4-DB	1.67	0.70	0.0117
Endrin	5.00	0.33	0.0167
Malathion	1.67	0.60	0.0100
Ethyl parathion	1.67	0.25	0.0042
Picloram	1.67	1.00	0.0167
PMA	1.67	0.15	0.0025
CONNECTICUT—2 SITES			
Atrazine	50.00	2.50	1.2500
DELAWARE—3 SITES			
Captan	33.33	0.04	0.0133
Lindane	33.33	0.08	0.0267
FLORIDA—15 SITES			
Atrazine	6.67	0.80	0.0533
Azinphosmethyl	6.67	2.50	0.1667
Captan	6.67	7.50	0.5000
Carbophenothion	6.67	2.00	0.1333
Chlorobenzilate	13.33	1.31	0.1753
Copper oxide	6.67	7.50	0.5000
Copper oxychloride sulfate	6.67	8.00	0.5333
2,4-D	6.67	1.50	0.1000
Dalapon	6.67	1.70	0.1133
DDT technical	6.67	7.00	0.4667
Diazinon	6.67	2.90	0.1933
Dichloropropene	6.67	194.40	12.9600
Dicofol	6.67	1.50	0.1000
Ethion	13.33	2.75	0.3667
Ferbam	6.67	9.12	0.6080
Mirex	6.67	0.01	0.0007
Ethyl parathion	13.33	2.85	0.3800
Methyl parathion	6.67	10.00	0.6667
Sulfur	6.67	46.50	3.1000
2,4,5-T	6.67	0.75	0.0500
TDE technical	6.67	10.00	0.6667
Toxaphene	6.67	2.00	0.1333
Zineb	13.33	6.55	0.8733
GEORGIA—28 SITES			
Atrazine	7.14	3.00	0.2143
Azodrin	3.57	4.00	0.1429
Benefin	7.14	1.12	0.0800
Captan	39.29	0.08	0.0307
Ceresan red	10.71	0.01	0.0011
Copper oxychloride sulfate	3.57	1.36	0.0486
Copper sulfate	3.57	2.72	0.0971
2,4-D	10.71	0.50	0.0536
DDT technical	21.43	14.18	3.0396
Disulfoton	3.57	2.00	0.0714
Folex	3.57	1.50	0.0536
Malathion	21.43	0.34	0.0732
Maleic hydrazide	7.14	2.41	0.1725
Methoxychlor	28.57	0.02	0.0046
Mirex	3.57	0.01	0.0004
Ethyl parathion	3.57	1.00	0.0357
Methyl parathion	14.29	1.84	0.2632
PCNB	3.57	10.00	0.3571
Sulfur	7.14	40.20	2.8714
Thiram	10.71	0.04	0.0043
Toxaphene	17.86	14.45	2.5804
Trifluralin	7.14	1.25	0.0893

TABLE 10.—*Summary of pesticides used in FY 1969 on cropland by State*—Continued

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
IDAHO—33 SITES			
Captan	12.12	0.01	0.0015
Ceresan M	6.06	0.01	0.0006
Ceresan L	15.15	0.01	0.0015
CIPC	3.03	2.00	0.0606
2,4-D	12.12	2.12	0.2576
2,4-DB	3.03	0.50	0.0152
DDT technical	6.06	0.50	0.0303
Dieldrin	3.03	0.01	0.0003
Diquat	3.03	0.83	0.0252
EPTC	3.03	0.38	0.0115
Hexachlorobenzene	3.03	0.01	0.0003
Ro-Neet	6.06	1.87	0.1136
Trifluralin	6.06	0.56	0.0342
ILLINOIS—141 SITES			
Aldrin	19.15	1.52	0.2914
Amiben	7.80	0.91	0.0707
Atrazine	9.22	2.19	0.2023
Captan	49.65	0.06	0.0317
Carbaryl	0.71	4.80	0.0340
CDAA	7.80	1.51	0.1176
Ceresan red	0.71	0.01	0.0001
Ceresan L	0.71	0.06	0.0004
Chevron RE-5353	1.42	2.56	0.0363
2,4-D	20.57	0.42	0.0864
2,4-DB	2.13	0.35	0.0075
Diazinon	3.55	1.86	0.0659
Dieldrin	2.13	0.23	0.0050
Heptachlor	9.93	0.46	0.0455
Linuron	1.42	0.66	0.0094
Malathion	39.72	0.03	0.0104
Methoxychlor	10.64	0.01	0.0011
Ramrod	7.80	1.22	0.0955
Roundup	0.71	0.07	0.0005
Silvex	0.71	0.25	0.0018
Thiram	0.71	0.01	0.0001
Trifluralin	2.84	0.97	0.0277
Vernolate	0.71	0.37	0.0026
INDIANA—75 SITES			
Aldrin	10.67	1.11	0.1187
Amiben	5.33	0.85	0.0453
Atrazine	13.33	1.79	0.2393
Captan	26.67	0.01	0.0027
Carbaryl	1.33	1.60	0.0213
CDAA	1.33	1.07	0.0143
Ceresan L	2.67	0.01	0.0003
2,4-D	10.67	0.35	0.0373
DDT technical	1.33	0.01	0.0001
Dieldrin	1.33	0.01	0.0001
Difolatan	1.33	0.01	0.0001
Heptachlor	5.33	0.32	0.0172
Malathion	17.33	0.01	0.0017
Methoxychlor	4.00	0.01	0.0004

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
INDIANA—75 SITES—Continued			
Methylmercury dicyandiamide	2.67	0.01	0.0003
Ramrod	2.67	1.40	0.0373
Roundup	1.33	1.50	0.0200
Trifluralin	2.67	0.75	0.0200
Zineb	1.33	1.60	0.0213
IOWA—151 SITES			
Aldrin	8.61	0.68	0.0587
Amiben	8.61	1.12	0.0966
Atrazine	10.60	2.00	0.2123
Captan	2.65	0.03	0.0008
Carbaryl	0.66	1.00	0.0066
CDAA	1.32	1.50	0.0199
Chevron RE-5353	0.66	1.00	0.0066
2,4-D	20.53	0.62	0.1278
Diazinon	6.62	1.19	0.0791
Dicamba	0.66	0.50	0.0033
Dieldrin	0.66	0.15	0.0010
Heptachlor	4.64	0.35	0.0164
Lindane	1.32	0.06	0.0009
Ethyl parathion	0.66	0.32	0.0021
Phorate	1.32	0.95	0.0126
Ramrod	3.97	2.02	0.0801
Randox T	0.66	0.40	0.0026
Thiram	0.66	0.06	0.0004
Trifluralin	2.65	0.47	0.0125
KENTUCKY—31 SITES			
Aldrin	9.68	2.00	0.1935
Atrazine	19.35	1.33	0.2581
2,4-D	3.23	0.50	0.0161
Dalapon	3.23	1.50	0.0484
DDT technical	3.23	3.00	0.0968
EPTC	3.23	1.50	0.0484
LOUISIANA—27 SITES			
Aldrin	22.22	0.08	0.0178
Captan	3.70	0.25	0.0093
Carbaryl	3.70	12.00	0.4444
Ceresan L	3.70	0.01	0.0004
Cotoran	3.70	1.00	0.0370
2,4-D	11.11	1.58	0.1759
Dalapon	3.70	2.00	0.0741
DDT technical	7.41	23.25	1.7222
DEF	3.70	9.00	0.3333
Dimetan	3.70	0.01	0.0004
Malathion	3.70	1.00	0.0370
Methylmercury dicyandiamide	3.70	0.01	0.0004
Methylmercury nitrile	3.70	0.01	0.0004
MSMA	3.70	1.50	0.0556
Nitrate	22.22	72.00	16.0000

TABLE 10.—*Summary of pesticides used in FY 1969 on cropland by State*—Continued

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
LOUISIANA—27 SITES—Continued			
Methyl parathion	7.41	7.20	0.5333
Propanil	11.11	3.17	0.3519
Silvex	3.70	1.00	0.0370
Strobane	3.70	18.00	0.6667
TCA	3.70	2.00	0.0741
Toxaphene	3.70	75.00	2.7778
Trifluralin	3.70	1.00	0.0370
MAINE—8 SITES			
Dalapon	12.50	4.90	0.6125
Dinitrobutylphenol	37.50	1.37	0.5125
Disulfoton	25.00	8.50	2.1250
Malathion	12.50	1.00	0.1250
Maneb	12.50	0.70	0.0875
Sodium arsenite	25.00	8.80	2.2000
MARYLAND—13 SITES			
Atrazine	30.77	1.26	0.3885
Captan	30.77	0.03	0.0100
2,4-D	15.38	0.54	0.0838
Dieldrin	7.69	0.01	0.0008
Lindane	15.38	0.01	0.0015
Malathion	23.08	0.01	0.0023
Methoxychlor	7.69	0.01	0.0008
Thiram	7.69	0.01	0.0008
MASSACHUSETTS—2 SITES			
Carbaryl	50.00	0.83	0.4150
Dinitrobutylphenol	50.00	3.06	1.5300
Disulfoton	50.00	1.50	0.7500
Dithane M-45	50.00	12.40	6.2000
Maleic hydrazide	50.00	2.32	1.1600
Oxydemetonmethyl	50.00	0.25	0.1250
Ethyl parathion	50.00	0.53	0.2650
MICHIGAN—51 SITES			
Atrazine	11.76	1.49	0.1753
Azinphosmethyl	1.96	8.00	0.1569
Captan	1.96	0.01	0.0002
CDAA	1.96	6.00	0.1176
Ceresan red	1.96	0.01	0.0002
CIPC	1.96	1.00	0.0196
Chloroxuron	3.92	2.63	0.1029
2,4-D	9.80	0.53	0.0524
DDT technical	1.96	1.50	0.0294
Dinitrobutylphenol	1.96	11.25	0.2206
Diuron	1.96	2.00	0.0392
EPTC	1.96	2.00	0.0392
Herbisan	1.96	10.00	0.1961
Malathion	3.92	0.50	0.0198
Methoxychlor	1.96	0.01	0.0002

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
MISSISSIPPI—29 SITES			
Azinphosmethyl	3.45	0.25	0.0086
Azodrin	6.90	0.76	0.0524
Bidrin	6.90	0.03	0.0024
Captan	24.14	0.09	0.0210
Ceresan M	3.45	0.01	0.0003
Ceresan red	27.59	0.02	0.0045
Ceresan L	3.45	0.01	0.0003
Chloroneb	17.24	0.06	0.0100
Cotoran	13.79	0.47	0.0652
DDT technical	31.03	3.47	1.0759
DEF	20.69	0.82	0.1690
Disulfoton	31.03	0.05	0.0152
Diuron	17.24	0.63	0.1079
DSMA	10.34	0.70	0.0728
Endrin	3.45	2.00	0.0690
Linuron	3.45	0.42	0.0145
Malathion	6.90	1.40	0.0969
Methoxychlor	3.45	0.01	0.0003
Mirex	6.90	0.01	0.0007
MSMA	10.34	1.44	0.1486
Norea	3.45	0.33	0.0114
Nitralin	13.79	0.99	0.1372
Methyl parathion	41.38	2.14	0.8841
PCNB	10.34	0.13	0.0131
Sodium chlorate	3.45	6.00	0.2069
Toxaphene	34.48	7.50	2.5862
Trifluralin	37.93	0.85	0.3241
Vernolate	3.45	0.30	0.0103
MISSOURI—81 SITES			
Aldrin	4.94	1.65	0.0815
Amiben	4.94	1.15	0.0568
Atrazine	12.35	1.87	0.2315
Bidrin	1.23	0.10	0.0012
Captan	1.23	0.01	0.0001
Ceresan M	1.23	0.01	0.0001
2,4-D	11.11	0.77	0.0856
2,4-DB	2.47	0.25	0.0062
Diazinon	1.23	0.93	0.0115
Dinitrobutylphenol	2.47	0.53	0.0131
Heptachlor	1.23	0.19	0.0023
Linuron	2.47	0.37	0.0091
NPA	2.47	1.07	0.0264
Methyl parathion	1.23	0.50	0.0062
Propazine	1.23	2.00	0.0247
Ramrod	1.23	1.10	0.0136
Trifluralin	7.41	0.91	0.0673
Vernolate	2.47	1.38	0.0340
NEBRASKA—103 SITES			
Amiben	0.97	2.50	0.0243
Atrazine	4.85	0.82	0.0400
Captan	17.48	0.04	0.0069
Ceresan red	0.97	0.01	0.0001

TABLE 10.—*Summary of pesticides used in FY 1969 on cropland by State*—Continued

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
NEBRASKA—103 SITES—Continued			
Ceresan L	0.97	0.01	0.0001
Chevron RE-5353	1.94	1.25	0.0243
2,4-D	14.56	0.44	0.0644
Diazinon	4.85	0.98	0.0476
Dieldrin	3.88	0.01	0.0004
Disulfoton	0.97	0.22	0.0021
EPTC	0.97	3.00	0.0291
Malathion	17.48	0.01	0.0017
Methoxychlor	1.94	0.01	0.0002
Methylmercury dicyandiamide	4.85	0.01	0.0005
Nabam	0.97	0.01	0.0001
Norea	0.97	0.60	0.0058
Ethyl parathion	3.88	0.50	0.0194
Phorate	0.97	0.90	0.0087
Ramrod	0.97	0.83	0.0081
Thiram	4.85	0.03	0.0014
NEW JERSEY—5 SITES			
2,4-D	40.00	0.31	0.1240
Monuron	20.00	1.60	0.3200
Ethyl parathion	20.00	0.54	0.1080
Sulfur	20.00	9.00	1.8000
NEW MEXICO—10 SITES			
Azodrin	10.00	1.50	0.1500
Carbaryl	10.00	2.50	0.2500
DDT technical	10.00	1.00	0.1000
Diuron	20.00	1.12	0.2250
Ethyl parathion	10.00	2.50	0.2500
Toxaphene	10.00	1.00	0.1000
NEW YORK—38 SITES			
Atrazine	23.68	1.36	0.3211
Azinphosmethyl	5.26	1.15	0.0605
Captan	13.16	0.87	0.1150
Carbaryl	5.26	1.55	0.0816
Copper sulfate	2.63	3.00	0.0789
2,4-D	7.89	0.21	0.0166
Dalapon	2.63	2.50	0.0658
DDT technical	5.26	1.35	0.0711
Demeton	2.63	0.04	0.0011
Diazinon	2.63	1.00	0.0263
Dichlone	2.63	2.20	0.0579
Dinitrobutylphenol	5.26	15.22	0.8013
Diuron	5.26	2.40	0.1263
Endosulfan (I)	5.26	0.95	0.0500
Lead arsenate	2.63	3.80	0.1000
Malathion	5.26	0.01	0.0005
MCPA	5.26	0.33	0.0176
Methoxychlor	2.63	0.01	0.0003
Methylmercury dicyandiamide	10.53	0.01	0.0011
Nabam	2.63	2.40	0.0632
Nitrate	7.89	26.17	2.0658
Oxydemetonmethyl	2.63	0.15	0.0039
Ethyl parathion	5.26	0.45	0.0239
Phosphamidon	2.63	0.15	0.0039
Sodium arsenite	2.63	0.90	0.0237
NORTH CAROLINA—29 SITES			
Aldrin	6.90	1.75	0.1207
Atrazine	6.90	2.75	0.1897
Carbaryl	20.69	1.43	0.2966
Ceresan red	3.45	0.10	0.0034
Chromophon	3.45	0.15	0.0052
Copper carbonate	3.45	0.60	0.0207
Copper-8-quinolinolate	3.45	0.01	0.0003
2,4-D	20.69	1.00	0.2079
2,4-DB	3.45	0.07	0.0024
DDT technical	13.79	0.70	0.0962
Diazinon	13.79	1.12	0.1545
Dicamba	3.45	1.20	0.0414
Dichloropropene	3.45	20.00	0.6897
Dieldrin	6.90	1.25	0.0866
Dinitrobutylphenol	3.45	1.50	0.0517
Diphenamid	6.90	1.07	0.0741
EPTC	3.45	4.00	0.1379
Ethylene dibromide	3.45	6.00	0.2069
Lindane	3.45	0.01	0.0003
Maleic hydrazide	10.34	0.47	0.0486
Ethyl parathion	6.90	0.50	0.0345
Methyl parathion	3.45	0.83	0.0286
Phorate	6.90	1.13	0.0783
Sulfur	3.45	11.10	0.3828
TDE technical	10.34	0.26	0.0272
Thiram	6.90	0.01	0.0007
Toxaphene	6.90	8.65	0.5966
Trifluralin	3.45	0.57	0.0197
Vernolate	6.90	1.85	0.1276
NORTH DAKOTA—159 SITES			
Barban	1.26	0.17	0.0021
Captan	0.63	0.01	0.0001
Ceresan M	0.63	0.01	0.0001
Ceresan red	2.52	0.01	0.0003
Ceresan L	5.03	0.01	0.0005
2,4-D	42.14	0.40	0.1673
Dicamba	1.89	0.08	0.0016
Disulfoton	0.63	3.00	0.0189
Endrin	0.63	0.25	0.0016
Heptachlor	1.26	0.04	0.0005
Lindane	1.89	0.02	0.0004
Malathion	0.63	0.01	0.0001
Maneb	0.63	1.50	0.0094
MCPA	5.66	0.30	0.0171
Methylmercury dicyandiamide	41.51	0.01	0.0042

TABLE 10.—*Summary of pesticides used in FY 1969 on cropland by State*—Continued

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
NORTH DAKOTA—159 SITES—Continued			
Phenylmercury urea	0.63	0.01	0.0001
PMA	1.26	0.01	0.0001
Polyram	0.63	10.40	0.0654
OHIO—66 SITES			
Aldrin	6.06	3.00	0.1818
Amiben	3.03	1.75	0.0530
Atrazine	12.12	1.20	0.1455
Captan	12.12	0.02	0.0021
Ceresan M	1.52	0.01	0.0002
Copper sulfate	1.52	1.60	0.0242
2,4-D	19.70	0.44	0.0859
Dalapon	1.52	1.50	0.0227
Diazinon	1.52	0.50	0.0076
Dichlone	1.52	1.80	0.0273
Dieldrin	1.52	0.01	0.0002
Dinocap	1.52	0.01	0.0002
Dithane M-45	1.52	0.30	0.0045
Linuron	1.52	0.75	0.0114
Malathion	10.61	0.01	0.0011
Maneb	3.03	0.75	0.0227
Methylmercury dicyandiamide	1.52	0.05	0.0008
NPA	1.52	2.27	0.0344
PCP	1.52	1.50	0.0227
Picloram	1.52	0.25	0.0038
Randox T	1.52	1.40	0.0212
Sulfur	1.52	25.00	0.3788
TDE technical	1.52	0.80	0.0121
Trifluralin	1.52	1.00	0.0152
Ziram	1.52	0.80	0.0121
OKLAHOMA—65 SITES			
Cacodylic acid	1.54	0.01	0.0002
Captan	4.62	0.01	0.0005
Carbaryl	1.54	0.30	0.0046
Ceresan M	20.00	0.01	0.0020
Ceresan red	12.31	0.01	0.0012
Chloroneb	1.54	0.01	0.0002
2,4-D	6.15	0.86	0.0531
2,4-DB	4.62	0.50	0.0231
Dieldrin	4.62	0.01	0.0005
Dimethoate	1.54	0.50	0.0077
Dinitrobutylphenol	1.54	2.00	0.0308
Disulfoton	7.69	0.58	0.0445
Falone	1.54	2.00	0.0308
Methylmercury dicyandiamide	1.54	0.01	0.0002
Nitrate	10.77	16.64	1.7923
Ethyl parathion	3.08	0.75	0.0231
Methyl parathion	12.31	0.65	0.0800
PCNB	1.54	0.01	0.0002
Phosphamidon	1.54	0.12	0.0018
Thiram	1.54	0.01	0.0002
Trifluralin	4.62	1.10	0.0508

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
PENNSYLVANIA—31 SITES			
Atrazine	19.35	1.57	0.3032
Azinphosmethyl	3.23	0.50	0.0161
Captan	3.23	0.01	0.0003
Carbaryl	3.23	0.32	0.0103
Chlordane	3.23	1.20	0.0387
Copper sulfate	3.23	1.70	0.0548
2,4-D	16.13	0.92	0.1484
DDT technical	9.68	0.83	0.0806
Dicofol	3.23	0.42	0.0135
Dinitrobutylphenol	3.23	0.82	0.0265
Dinitrocresol	3.23	3.00	0.0968
Dinocap	3.23	0.44	0.0142
Diuron	3.23	0.32	0.0103
Lindane	3.23	0.02	0.0006
Linuron	3.23	1.00	0.0323
Maneb	3.23	7.00	0.2258
Methyl demeton	3.23	1.50	0.0484
Nitrate	3.23	100.00	3.2258
Ethyl parathion	6.45	0.45	0.0290
Phorate	3.23	12.50	0.4032
Simazine	3.23	0.40	0.0129
Sodium arsenite	3.23	2.50	0.0806
Trifluralin	3.23	0.75	0.0242
RHODE ISLAND—1 SITE			
Carbaryl	100.00	0.80	0.8000
DDT technical	100.00	2.00	2.0000
Disulfoton	100.00	2.00	2.0000
Dithane M-45	100.00	6.40	6.4000
EPTC	100.00	5.00	5.0000
Oxydemetonmethyl	100.00	0.80	0.8000
SOUTH CAROLINA—17 SITES			
Azodrin	5.88	0.40	0.0235
Carbaryl	17.65	7.19	1.2682
2,4-D	11.76	0.40	0.0471
DDT technical	29.41	2.46	0.7229
DEF	5.88	0.20	0.0118
Demeton	5.88	1.60	0.0941
Diuron	5.88	0.72	0.0424
MSMA	5.88	0.45	0.0265
Nabam	5.88	1.20	0.0706
Ethyl parathion	11.76	0.51	0.0600
Methyl parathion	11.76	5.10	0.6000
Phorate	5.88	0.20	0.0118
TDE technical	5.88	2.25	0.1324
Toxaphene	17.65	6.17	1.0894
Trifluralin	35.29	0.21	0.0753
SOUTH DAKOTA—106 SITES			
Atrazine	1.89	1.40	0.0264
Captan	10.38	0.01	0.0010
Carbaryl	0.94	1.05	0.0099

TABLE 10.—*Summary of pesticides used in FY 1969 on cropland by State*—Continued

Compound	Percent of Sites Treated	Average Application Rate (lb/acre)	Average Amount Applied Per Site (lb/acre)
SOUTH DAKOTA—106 SITES—Continued			
Ceresan M	0.94	0.01	0.0001
2,4-D	26.42	0.47	0.1230
Dalapon	0.94	0.74	0.0070
Dieldrin	1.89	0.01	0.0002
Heptachlor	3.77	0.02	0.0007
Lindane	0.94	0.01	0.0001
Malathion	6.60	0.01	0.0007
MCPA	4.72	0.20	0.0095
Methoxychlor	3.77	0.01	0.0004
Methylmercury dicyandiamide	10.38	0.01	0.0010
Phorate	0.94	0.60	0.0057
Ramrod	0.94	1.00	0.0094
Thiram	0.94	0.01	0.0001
TENNESSEE—28 SITES			
Atrazine	21.43	1.98	0.4250
Bidrin	3.57	0.54	0.0193
Captan	10.71	0.01	0.0011
Ceresan M	7.14	0.01	0.0007
Ceresan red	3.57	0.01	0.0004
Cotoran	7.14	0.78	0.0557
2,4-DB	7.14	0.43	0.0307
Disulfoton	3.57	0.01	0.0004
Diuron	7.14	0.06	0.0046
Linuron	7.14	0.75	0.0536
Malathion	3.57	0.01	0.0004
Methylmercury dicyandiamide	3.57	0.01	0.0004
MSMA	3.57	0.46	0.0164
Nitrate	7.14	250.00	17.8571
Nitralin	3.57	0.15	0.0054
PCNB	3.57	0.01	0.0004
Trifluralin	14.29	0.38	0.0543
UTAH—12 SITES			
Dichloropropene	8.33	180.00	15.0000
Heptachlor	8.33	0.34	0.0283
VIRGINIA—20 SITES			
Atrazine	5.00	4.00	0.2000
Azinphosmethyl	5.00	2.00	0.1000
Carbaryl	5.00	2.75	0.1375
Copper oxide	10.00	2.60	0.2600
2,4-D	10.00	1.12	0.1125
2,4-DB	5.00	0.20	0.0100
DDT technical	5.00	2.00	0.1000
Diazinon	5.00	0.50	0.0250
Dinitrobutylphenol	5.00	1.50	0.0750
Diphenamid	5.00	4.00	0.2000
Disulfoton	10.00	6.80	0.6800
Ethylene dibromide	5.00	23.24	1.1620
Dichloropropane	5.00	54.43	2.7215
Malathion	5.00	0.95	0.0475
Methoxychlor	5.00	1.00	0.0500
Ethyl parathion	5.00	6.00	0.3000
Phorate	5.00	3.00	0.1500
Sulfur	5.00	57.00	2.800
Vernolate	5.00	2.40	0.1200
WASHINGTON—2 SITES			
Ceresan L	50.00	0.01	0.0050
2,4-D	50.00	1.00	0.5000
WEST VIRGINIA—5 SITES			
Azinphosmethyl	20.00	0.50	0.1000
Ethyl parathion	20.00	1.50	0.3000
WISCONSIN—68 SITES			
Atrazine	29.41	2.61	0.7684
Ceresan red	1.47	0.01	0.0001
2,4-D	2.94	0.75	0.0221
Ramrod	1.47	2.00	0.0294
Trifluralin	1.47	2.00	0.0294

NOTE: Of the 43 States with cropland soil analyzed, use records for 4 showed no pesticides used on the sampling sites: Nevada (2 sites); New Hampshire (2 sites); Vermont (5 sites); and Wyoming (17 sites).

TABLE 11.—*Summary of pesticides used in FY 1969 on noncropland, by State*

Compound	Percent of Sites Treated	Average Application Rate Per Site Treated (lb/acre)	Average Amount Applied Per Site (lb/acre)
GEORGIA—15 SITES			
Mirex	6.67	0.01	0.0007
IDAHO—26 SITES			
Malathion	3.85	0.61	0.0235
NEBRASKA—19 SITES			
2,4-D	5.26	2.00	0.1053

NOTE: Of the 11 States with noncropland soil analyzed, 8 reported no pesticides used on the sampling sites: Arizona (43 sites); Iowa (7 sites); Maine (11 sites); Maryland (3 sites); Virginia (14 sites); Washington (11 sites); West Virginia (9 sites); and Wyoming (37 sites).

TABLE 12.—*Comparison of residues detected with use records for 12 States with highest arsenic residues, FY 1969*

State	Average Amount Applied (lb/acre)	Percent of Sites Treated	Mean Residue Level (ppm)	Percent Positive Sites [1]
Arkansas	[3] 0.13	4.4	9.0	100.0
Kentucky	No Arsenic Compounds Used		8.4	100.0
New England [2]	[4] 0.88	10.0	10.2	100.0
New York	[5] 0.12	5.3	9.4	94.6
North Dakota	No Arsenic Compounds Used		8.5	100.0
Ohio	No Arsenic Compounds Used		11.2	100.0
Pennsylvania	[4] 0.08	3.2	10.8	100.0

[1] Percent based on number of sites with residues greater than or equal to the sensitivity limits.
[2] Connecticut, Maine, Massachusetts, New Hampshire, Rhode Island, and Vermont.
[3] Calculated for DSMA.
[4] Calculated for sodium arsenite.
[5] Calculated for sodium arsenite and lead arsenate.

TABLE 13.—*Comparison of residues detected with use records for 5 States with highest DDTR residues, FY 1969*

State	Average Amount Applied (lb/acre)	Percent of Sites Treated	Mean Residue Level (ppm)	Percent Positive Sites [1]
Alabama	4.20	39.1	1.13	90.9
California	0.38	13.6	1.47	84.6
Michigan	0.03	2.0	2.09	23.5
Mississippi	1.07	31.0	2.06	89.7
South Carolina	0.72	29.4	1.17	88.2

[1] Percent based on number of sites with residues greater than or equal to the sensitivity limits.

TABLE 14.—*Comparison of residues detected with use records for 7 States with highest dieldrin residues, FY 1969*

State	Average Amount Applied (lb/acre)	Percent of Sites Treated	Mean Residue Level (ppm)	Percent Positive Sites [1]
Florida	0.00	0.0	0.08	38.9
Illinois	0.29 aldrin	19.2		
	0.01 dieldrin	2.1	0.11	61.3
Iowa	0.06 aldrin	8.6		
	<0.01 dieldrin	0.7	0.06	53.6
Kentucky	0.19 aldrin	9.7	0.06	54.8
North Carolina	0.12 aldrin	6.9		
	0.09 dieldrin	6.9	0.08	32.3
Virginia/West Virginia	0.00	0.0	0.07	25.9

[1] Percent based on number of sites with residues greater than or equal to the sensitivity limits.

TABLE 15.—*Fiftieth percentile value for pesticide residues in cropland soil including the 95% confidence interval by State*

Pesticide	Upper Limit (ppm)	Fiftieth Percentile Residue Level (ppm)	Lower Limit (ppm)
ALABAMA			
Arsenic	4.42	4.09	3.76
p,p'-DDE	0.10	0.07	0.04
o,p'-DDT	0.03	0.02	0.01
p,p'-DDT	0.27	0.21	0.15
DDTR	0.48	0.38	0.29
p,p'-TDE	0.02	0.01	0.01
ARKANSAS			
Arsenic	7.42	7.26	7.10
p,p'-DDE	0.02	0.02	0.02
o,p'-DDT	0.01	0.01	0.00
p,p'-DDT	0.04	0.03	0.03
DDTR	0.10	0.09	0.08
Dieldrin	0.00	0.00	0.00
p,p'-TDE	0.01	0.01	0.01
Toxaphene	0.15	0.04	0.00
CALIFORNIA			
Arsenic	4.02	3.92	3.83
o,p'-DDE	0.00	0.00	0.00
p,p'-DDE	0.05	0.04	0.04
o,p'-DDT	0.01	0.01	0.00
p,p'-DDT	0.04	0.03	0.03
DDTR	0.14	0.13	0.12
Dieldrin	0.00	0.00	0.00
o,p'-TDE	0.00	0.00	0.00
p,p'-TDE	0.02	0.01	0.01
Toxaphene	0.02	0.01	0.00
COLORADO			
Arsenic	4.26	4.20	4.15
FLORIDA			
Arsenic	0.64	0.58	0.53
Chlordane	0.05	0.03	0.02
p,p'-DDE	0.03	0.02	0.01
o,p'-DDT	0.01	0.00	0.00
p,p'-DDT	0.07	0.05	0.03
DDTR	0.10	0.08	0.06
p,p'-TDE	0.02	0.01	0.00
GEORGIA			
Arsenic	1.96	1.88	1.80
p,p'-DDE	0.06	0.05	0.04
o,p'-DDT	0.01	0.01	0.01
p,p'-DDT	0.17	0.01	0.09
DDTR	0.30	0.23	0.17
p,p'-TDE	0.01	0.01	0.01
Toxaphene	0.36	0.28	0.18

Pesticide	Upper Limit (ppm)	Fiftieth Percentile Residue Level (ppm)	Lower Limit (ppm)
IDAHO			
Arsenic	2.85	2.53	2.21
p,p'-DDT	0.00	0.00	0.00
DDTR	0.01	0.00	0.00
ILLINOIS			
Aldrin	0.00	0.00	0.00
Arsenic	6.28	6.20	6.13
Chlordane	0.01	0.01	0.00
DDTR	0.00	0.00	0.00
Dieldrin	0.03	0.02	0.02
Heptachlor	0.00	0.00	0.00
Heptachlor epoxide	0.00	0.00	0.00
INDIANA			
Aldrin	0.00	0.00	0.00
Arsenic	7.24	7.03	6.82
Dieldrin	0.00	0.00	0.00
IOWA			
Aldrin	0.00	0.00	0.00
Arsenic	5.86	5.78	5.71
Atrazine	0.01	0.00	0.00
Chlordane	0.01	0.01	0.00
p,p'-DDE	0.00	0.00	0.00
p,p'-DDT	0.00	0.00	0.00
DDTR	0.00	0.00	0.00
Dieldrin	0.02	0.01	0.01
Heptachlor	0.00	0.00	0.00
Heptachlor epoxide	0.00	0.00	0.00
KENTUCKY			
Aldrin	0.00	0.00	0.00
Arsenic	8.45	7.89	7.30
Dieldrin	0.01	0.01	0.00
LOUISIANA			
Arsenic	1.80	1.65	1.51
p,p'-DDE	0.01	0.00	0.00
o,p'-DDT	0.01	0.00	0.00
p,p'-DDT	0.02	0.01	0.00
DDTR	0.02	0.01	0.01
Dieldrin	0.01	0.01	0.00
MICHIGAN			
Arsenic	4.92	3.83	2.93
p,p'-DDE	0.00	0.00	0.00
DDTR	0.00	0.00	0.00
Dieldrin	0.00	0.00	0.00

TABLE 15.—*Fiftieth percentile value for pesticide residues in cropland soil including the 95% confidence interval by State*—Continued

Pesticide	Upper Limit (ppm)	Fiftieth Percentile Residue Level (ppm)	Lower Limit (ppm)
MID-ATLANTIC STATES GROUP [1]			
Arsenic	5.87	5.34	4.83
MISSISSIPPI			
Arsenic	4.86	4.68	4.51
p,p'-DDE	0.11	0.09	0.08
o,p'-DDT	0.06	0.06	0.05
p,p'-DDT	0.36	0.29	0.24
DDTR	0.67	0.55	0.45
p,p'-TDE	0.03	0.02	0.01
Toxaphene	0.24	0.16	0.09
MISSOURI			
Aldrin	0.00	0.00	0.00
Arsenic	5.43	5.05	4.69
Dieldrin	0.00	0.00	0.00
NEBRASKA			
Arsenic	4.73	4.56	4.40
Chlordane	0.01	0.00	0.00
p,p'-DDE	0.00	0.00	0.00
DDTR	0.00	0.00	0.00
Dieldrin	0.00	0.00	0.00
NEW ENGLAND STATES GROUP [2]			
Arsenic	6.39	5.71	5.09
p,p'-DDE	0.02	0.01	0.00
p,p'-DDT	0.05	0.02	0.00
DDTR	0.04	0.02	0.01
p,p'-TDE	0.01	0.01	0.00
NEW YORK			
Arsenic	6.34	6.12	5.90
p,p'-DDE	0.00	0.00	0.00
o,p'-DDT	0.00	0.00	0.00
p,p'-DDT	0.01	0.00	0.00
DDTR	0.00	0.00	0.00
Dieldrin	0.00	0.00	0.00
p,p'-TDE	0.00	0.00	0.00
NORTH CAROLINA			
Arsenic	3.42	3.07	2.77
p,p'-DDE	0.03	0.03	0.02
o,p'-DDT	0.02	0.01	0.01
p,p'-DDT	0.05	0.03	0.02
DDTR	0.18	0.13	0.08
Dieldrin	0.01	0.00	0.00
o,p'-TDE	0.03	0.02	0.01
NORTH CAROLINA—Continued			
p,p'-TDE	0.03	0.02	0.01
Toxaphene	0.16	0.07	0.01
NORTH DAKOTA			
Arsenic	6.82	6.79	6.75
p,p'-DDT	0.00	0.00	0.00
DDTR	0.00	0.00	0.00
OHIO			
Aldrin	0.00	0.00	0.00
Arsenic	7.85	7.58	7.32
DDTR	0.00	0.00	0.00
Dieldrin	0.00	0.00	0.00
OKLAHOMA			
Arsenic	2.19	2.11	2.02
PENNSYLVANIA			
Arsenic	7.22	6.65	6.15
p,p'-DDE	0.00	0.00	0.00
p,p'-DDT	0.00	0.00	0.00
DDTR	0.00	0.00	0.00
Dieldrin	0.01	0.00	0.00
p,p'-TDE	0.00	0.00	0.00
SOUTH CAROLINA			
Arsenic	1.98	1.82	1.68
p,p'-DDE	0.08	0.06	0.03
o,p'-DDT	0.04	0.03	0.02
p,p'-DDT	0.18	0.13	0.08
DDTR	0.31	0.15	0.06
p,p'-TDE	0.06	0.04	0.02
SOUTH DAKOTA			
Arsenic	3.86	3.76	3.68
Dieldrin	0.00	0.00	0.00
TENNESSEE			
Arsenic	7.18	7.00	6.81
p,p'-DDE	0.00	0.00	0.00
p,p'-DDT	0.01	0.00	0.00
DDTR	0.01	0.01	0.00

TABLE 15.—*Fiftieth percentile value for pesticide residues in cropland soil including the 95% confidence interval by State*—Continued

Pesticide	Upper Limit (ppm)	Fiftieth Percentile Residue Level (ppm)	Lower Limit (ppm)
VIRGINIA AND WEST VIRGINIA			
Arsenic	2.90	2.72	2.56
p,p'-DDT	0.01	0.00	0.00
DDTR	0.02	0.01	0.01
Heptachlor epoxide	0.01	0.00	0.00
WASHINGTON			
Arsenic	2.30	2.22	2.14
p,p'-DDT	0.00	0.00	0.00
DDTR	0.00	0.00	0.00
WESTERN STATES GROUP [3]			
Arsenic	3.57	3.40	3.23
p,p'-DDE	0.01	0.00	0.00
p,p'-DDT	0.00	0.00	0.00
DDTR	0.01	0.01	0.00
WISCONSIN			
Arsenic	3.33	3.14	2.97
DDTR	0.00	0.00	0.00
Dieldrin	0.00	0.00	0.00
WYOMING			
Arsenic	0.92	0.85	0.78

[1] Includes Delaware, Maryland, and New Jersey.
[2] Includes Connecticut, Maine, Massachusetts, New Hampshire, Rhode Island, and Vermont.
[3] Includes Arizona, Nevada, New Mexico, and Utah.

TABLE 16.—*Mean pesticide residues in ppm in soil for various cropping regions, FY 1969*

Compound	Corn	Cotton	Cotton and General Farming	General Farming	Hay and General Farming	Irrigated Land	Small Grains	Vegetable	Vegetable and Fruit
Aldrin	0.05	<0.01	<0.01	0.02	<0.01	<0.01	<0.01	<0.01	0.01
Arsenic	7.44	6.72	4.88	5.35	6.42	4.77	5.70	8.75	3.27
Atrazine	0.02						<0.01		
Carbophenothion			—	—		—			
Chlordane	0.09	<0.01	0.01	0.01	0.03	0.03	<0.01	<0.01	0.14
2,4-D				<0.01			<0.01	—	
DCPA	—	—	—	—	—	<0.01	—	—	—
o,p'-DDE	<0.01	0.01	<0.01	<0.01	<0.01	0.01	—	<0.01	0.01
p,p'-DDE	0.01	0.16	0.13	0.07	0.05	0.18	<0.01	0.18	0.37
o,p'-DDT	0.01	0.09	0.04	0.05	0.03	0.05	<0.01	0.07	0.06
p,p'-DDT	0.06	0.54	0.22	0.25	0.20	0.19	<0.01	0.50	0.64
DDTR	0.14	0.87	0.44	0.43	0.30	0.48	<0.01	0.81	1.92
DEF	—	—	—	<0.01	—	—	—	—	—
Diazinon		—	—			0.01			
Dicofol	<0.01	—	—	—	<0.01	0.01	—	—	—
Dieldrin	0.05	0.01	<0.01	0.03	0.02	0.02	<0.01	0.05	0.04
Endosulfan (I)	<0.01	—	—	—	<0.01	<0.01	—	—	—
Endosulfan (II)	<0.01	—	—	—	<0.01	0.01	—	<0.01	—
Endosulfan sulfate	<0.01	—	—	—	<0.01	0.01	—	<0.01	—
Endrin	<0.01	0.01	<0.01	<0.01	—	0.01	<0.01	0.01	0.01
Endrin aldehyde	—	—	—	—	—	—	—	—	<0.01
Endrin ketone	—	<0.01	<0.01	—	—	<0.01	—	<0.01	<0.01
Ethion		—	—			<0.01			
Ethyl parathion		—	—	<0.01	—	<0.01			
Heptachlor	0.01	—	<0.01	<0.01	<0.01	<0.01	—	—	<0.01
Heptachlor epoxide	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Isodrin	<0.01	—	—	<0.01	—	—	—	—	—
Lindane	<0.01	—	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	—
Malathion		—	—			—			
Methoxychlor	—	—	—	—	—	—	—	<0.01	—
PCNB	—	—	—	<0.01	—	—	—	—	—
o,p'-TDE	<0.01	<0.01	<0.01	0.01	<0.01	0.01	—	0.01	0.15
p,p'-TDE	0.05	0.07	0.04	0.04	0.01	0.04	<0.01	0.05	0.70
Toxaphene	<0.01	0.42	0.20	0.16	—	0.14	—	0.01	0.08
Trifluralin	<0.01	0.01	<0.01	<0.01	—	0.01	<0.01	<0.01	<0.01

NOTE: Blank = not analyzed; — = not detected.

TABLE 17.—*Percent of sites with detectable pesticide residues in ppm in soil for various cropping regions, FY 1969*

Compound	Corn	Cotton	Cotton and General Farming	General Farming	Hay and General Farming	Irrigated Land	Small Grains	Vegetable	Vegetable and Fruit
Aldrin	23.5	6.4	0.9	6.6	2.1	6.5	0.6	1.1	3.0
Arsenic	100.0	100.0	98.4	99.4	99.3	99.1	99.4	98.9	93.9
Atrazine	14.5						8.3		
Carbophenothion			—	—	—	—			
Chlordane	14.5	1.8	2.6	7.2	5.5	11.1	0.9	4.3	21.2
2,4-D				14.3			1.7	—	
DCPA	—	—	—	—	—	0.9	—	—	—
o,p'-DDE	0.5	15.6	5.2	10.8	2.8	19.4	—	5.3	15.1
p,p'-DDE	9.5	69.7	44.8	46.4	21.4	58.3	5.8	38.3	60.6
o,p'-DDT	3.0	51.4	25.0	27.1	10.3	33.3	3.0	23.4	36.4
p,p'-DDT	7.7	66.1	43.1	42.2	16.5	53.7	5.8	31.9	57.6
DDTR	10.9	72.5	47.4	49.4	22.8	60.2	6.1	39.4	63.6
DEF	—	—	—	0.6	—	—	—	—	—
Diazinon		—	—			12.5			
Dicofol	0.6	—	—	—	0.7	5.6	—	—	—
Dieldrin	41.8	24.8	14.7	25.3	15.2	39.8	7.0	23.4	21.2
Endosulfan (I)	0.3	—	—	—	0.7	1.8	—	—	—
Endosulfan (II)	0.2	—	—	—	0.7	5.6	—	1.1	—
Endosulfan sulfate	0.3	—	—	—	1.4	5.6	—	1.1	—
Endrin	0.3	7.3	2.6	3.6	—	11.1	0.9	3.2	6.1
Endrin aldehyde	—	—	—	—	—	—	—	—	3.0
Endrin ketone	—	2.8	0.9	—	—	2.8	—	1.1	3.0
Ethion		—	—			6.3			
Ethyl parathion		—	—	10.0	—	12.5			
Heptachlor	8.6	—	1.7	1.8	1.4	0.9	—	—	3.0
Heptachlor epoxide	13.3	0.9	3.4	7.8	4.1	13.0	0.9	4.3	12.1
Isodrin	1.2	—	—	1.8	—	—	—	—	—
Lindane	0.3	—	2.6	1.2	0.7	1.8	0.6	3.2	—
Malathion		—	—			—			
Methoxychlor	—	—	—	—	—	—	—	1.1	—
PCNB	—	—	—	0.6	—	—	—	—	—
o,p'-TDE	0.3	0.9	2.6	10.8	2.1	13.0	—	5.3	6.1
p,p'-TDE	3.3	47.7	25.9	35.5	11.7	38.0	1.8	27.7	45.4
Toxaphene	0.2	22.9	12.1	10.2	—	12.0	—	1.1	6.1
Trifluralin	2.0	12.8	7.8	6.0	—	9.3	0.3	2.1	3.0

NOTE: Blank = not analyzed; — = not detected.

BRIEFS

Residues of Chlorinated Hydrocarbon Pesticides in the Northern Quahog (Hard-Shell Clam), Mercenaria mercenaria*—1968 and 1969*

Ronald M. Check[1] and Manuel T. Canario, Jr.[2]

ABSTRACT

Samples of the northern quaghog (hard-shell clam), Mercenaria mercenaria, *were collected monthly, when possible, from September 1968 to September 1969 at five locations in Narragansett Bay, Rhode Island, and one location in nearby Mount Hope Bay. All 56 composite samples contained dieldrin at an average level of 0.040 ppm; p,p'-DDD was present in 3 samples at an average level of 0.026 ppm. Quahogs from upper reaches of Narragansett Bay contained higher levels of residues than samples from lower Bay areas.*

Introduction

Preliminary examinations of the northern quahog (hard-shell clam), *Mercenaria mercenaria,* revealed the presence of chlorinated hydrocarbon pesticides in samples from Narragansett Bay, Rhode Island. As a result, a one-year survey was conducted with sampling at monthly intervals, when possible, from September 1968-September 1969, to determine the amounts of various chlorinated pesticides present in this species.

Sampling and Analytical Procedures

In September 1968, sampling stations were established at five sites in upper Narragansett Bay and one station in the southeastern corner of nearby Mount Hope Bay (Fig. 1). Shellfish samples were obtained by dredging. In the laboratory, clams were shucked and drained; a 300-g composite sample of meat, generally representing 14-18 clams from each location, was blended in a Waring Blendor until homogenized. Blended samples were frozen until analyzed.

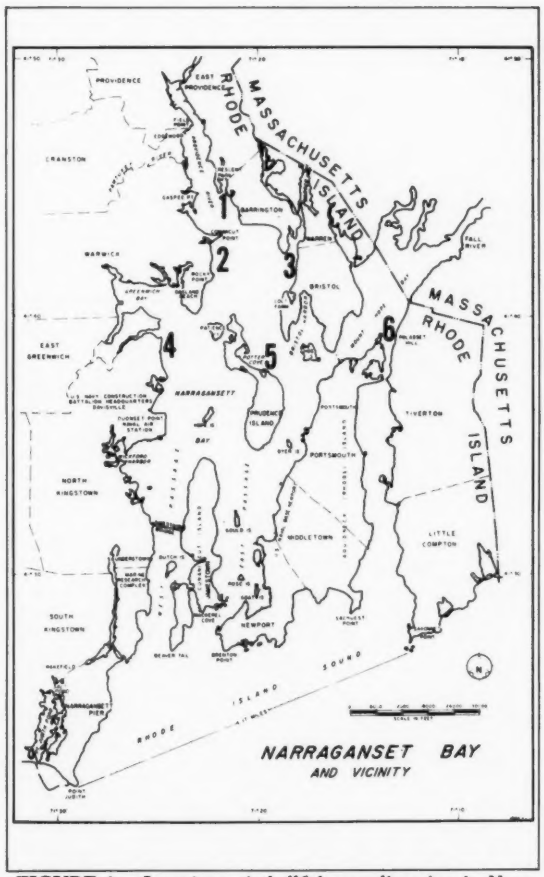

FIGURE 1.—*Locations of shellfish sampling sites in Narragansett Bay and Mount Hope Bay, R. I., September 1968-September 1969*

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The frozen 300-g samples were thawed, and 50-g subsamples were slurried with 100 ml of acetonitrile and allowed to stand overnight. A 25-g aliquot was filtered from the slurry and partitioned into petroleum ether according to the procedure outlined in the Pesticide Analytical Manual (*1*).

A 5-g aliquot was evaporated at room temperature to 2-3 ml and subjected to chemical cleanup by the sweep co-distillation method of Storherr *et al.* (*2*). The Florisil column step was included in the cleanup method. Purified sample extracts were concentrated to the equivalent of 1 g of sample per ml and analyzed by gas chromatography. The chromatograph was equipped with a Ni^{63} electron capture detector and two 6-ft glass columns, ¼ inch in diameter. One column contained 10% DC-200 and the other, 5% QF-1. These liquid phases were coated on Gas Chrom Q solid support. Nitrogen was used as the carrier gas. Injections onto the two columns provided tentative peak identification. All pesticide residues found were confirmed by thin layer chromatography using precoated aluminum oxide G plates. Standard solutions of the suspected pesticides were spotted beside samples and developed in *n*-heptane which had been redistilled over molten metallic sodium. Sample areas only were covered with aluminum foil, sprayed with Kovac's chromagenic reagent (*3*), and the plates were exposed to UV light. The foil was removed, and zones corresponding to standards were scraped from the plate and extracted from the aluminum oxide with 2 ml of 10% ethyl ether in petroleum ether. The 2-ml extracts were concentrated to 0.2 ml and reinjected through the two gas chromatographic columns for pesticide confirmation.

Further tests using chemical derivatization and partition coefficients were employed on initial samples to confirm the presence of dieldrin. The lower limits of detection for pesticides reported were 0.01 ppm for dieldrin and 0.02 ppm for *p,p'*-DDD, *p,p'*-DDE, and *p,p'*-DDT.

Occasional samples were fortified with known amounts of dieldrin, *o,p'*-DDE, *p,p'*-DDE, *p,p'*-DDD, and *p,p'*-DDT and subjected to the methodology described. Recoveries averaged 85%, 80%, 71%, 71%, and 73%, respectively. Ranges of recovery were 65%-100% for dieldrin and 50%-87% for *p,p'*-DDT. The recovery for *p,p'*-DDT and *p,p'*-DDE, while lower than that for dieldrin, was sufficient to show the presence of these compounds above the 0.02 ppm detection level used in this study. Several methods of analysis were evaluated initially including the traditional Florisil column cleanup procedure, but interfering peaks prevented positive identification and accurate quantitation of DDE and DDT if these compounds were present. Many of these peaks may have been residues of various polychlorinated biphenyls, and it was necessary to eliminate them before an accurate pesticide profile could be obtained. The co-distillation method provided a means of separating unknown peaks from pesticides present in the samples. Future monitoring programs in conjunction with currently available methodology should yield more information about possible PCB residues.

Results and Discussion

Analyses for chlorinated pesticides in quahogs taken from the sampling stations indicated in Fig. 1, revealed the presence of dieldrin in all 56 samples and *p,p'*-DDD in 3 samples (Table 1). The absence of DDE and DDT in amounts greater than 0.02 ppm was unexpected in the samples analyzed. The average levels of pesticides found during the year were 0.040 ppm for dieldrin and 0.026 ppm for *p,p'*-DDD. In general, residues of dieldrin and DDD were higher in those samples from upper reaches of the Narragansett Bay than from the lower Bay areas.

TABLE 1.—*Dieldrin and p,p'-DDD residues detected in northern quahogs, Narragansett and Mount Hope Bays—September 1968-September 1969*

Month of Sampling	Dieldrin and *p,p'*-DDD* Residues in PPM, Wet-Weight Basis, Drained Meat					
	Station 1	Station 2	Station 3	Station 4	Station 5	Station 6
1968						
Sept.	.060	.050	.010	.006	.021	.027
Oct.	.090	.056	.043	.063	.055	.048
1969						
Feb.	.070	.082	.043	.036	.037	.029
Mar.	086	.066	.045	.062	.015	.026
Apr.	.060	.063	.060	.049	.031	.024
May	.066	.050	.042			
June	.047	.047	.049		.039	.013
July	.044	.039	.053	.039	.029	.016
		*.028	*.030	*.020		
Aug.	.030	.023	.028	.014	.013	.007
Sept	.030	.026	.029	.026	.025	.023

NOTE: Blank = sample not analyzed.
* *P,p'*-DDD residues were detected only in the July samples from Stations 2, 3, and 4.

See Appendix for chemical names of compounds discussed in this paper.

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APPENDIX

Chemical Names of Compounds Discussed in This Issue

ALDRIN	Not less than 95% of 1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4-*endo-exo*-5,8-dimethanonaphthalene
ARSENIC	As_2O_3
ATRAZINE	2-chloro-4-ethylamino-6-isopropylamino-*s*-triazine
BHC	1,2,3,4,5,6-hexachlorocyclohexane, mixed isomers
CARBOPHENOTHION (TRITHION®)	*S*-[(*p*-chlorophenylthio)methyl] *0,0*-diethyl phosphorodithioate
CHLORDANE	1,2,4,5,6,7,8,8-octachloro-3a,4,7,7a-tetrahydro-4,7-methanoindane
2,4-D	2,4-dichlorophenoxyacetic acid
DCPA (DACTHAL®)	dimethyl ester of tetrachloroterephthalic acid
DDE	1,1-dichloro-2,2-bis(*p*-chlorophenyl) ethylene
DDT (including its isomers and dehydrochlorination products)	1,1,1-trichloro-2,2-bis(*p*-chlorophenyl)ethane; technical DDT consists of a mixture of the *p,p'*-isomer and the *o,p'*-isomer (in a ratio of about 3 or 4 to 1)
DIAZINON	*0,0*-diethyl *0*-(2-isopropyl-4-methyl-6-pyrimidyl) phosphorothioate
DCBP	4,4'-dichlorobenzophenone
DICOFOL (KELTHANE®)	4,4'-dichloro-*a*-(trichloromethyl)benzhydrol
DIELDRIN	Not less than 85% of 1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8a-octahydro-1,4-*endo-exo*-5,8-dimethano=naphthalene
DURSBAN®	*0,0*-diethyl *0*-(3,5,6-trichloro-2-pyridyl) phosphorothioate
ENDOSULFAN (THIODAN®)	6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin 3-oxide
ENDRIN	1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-*endo-endo*-5,8-dimethanonaphthalene
ETHION	*0,0,0',0'*-tetraethyl *S,S'*-methylenebisphorodithioate
HEPTACHLOR	1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methanoindene
HEPTACHLOR EPOXIDE	1,4,5,6,7,8,8-heptachloro-2,3-epoxy-3a,4,7,7a-tetrahydro-4,7-methanoindan
HCB	hexachlorobenzene
ISODRIN	1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4-*endo, endo*-5,8-dimethanonaphthalene
LINDANE	1,2,3,4,5,6-hexachlorocyclohexane, 99% or more gamma isomer
MALATHION	diethyl mercaptosuccinate, S-ester with 0,0-dimethyl phosphorodithioate
MERCURY	Hg
METHOXYCHLOR	1,1,1,-trichloro-2,2-bis(*p*-methoxyphenyl)ethane
METHYL MERCURY	R_2CH_3Hg
PARATHION	*0, 0*-diethyl *0*-*p*-nitrophenyl phosphorothioate
PCNB	pentachloronitrobenzene
POLYCHLORINATED BIPHENYLS (PCB's)	Mixtures of chlorinated biphenyl compounds having various percentages of chlorination
TDE (DDD) (including its isomers and dehydrochlorination products)	1,1-dichloro-2,2-bis(*p*-chlorophenyl)ethane; technical TDE contains some *o,p'*-isomer also
TOXAPHENE	chlorinated camphene containing 67% to 69% chlorine
TRIFLURALIN	*α,α,α*-trifluoro-2,6-dinitro-*N,N*-dipropyl-*p*-toluidine

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